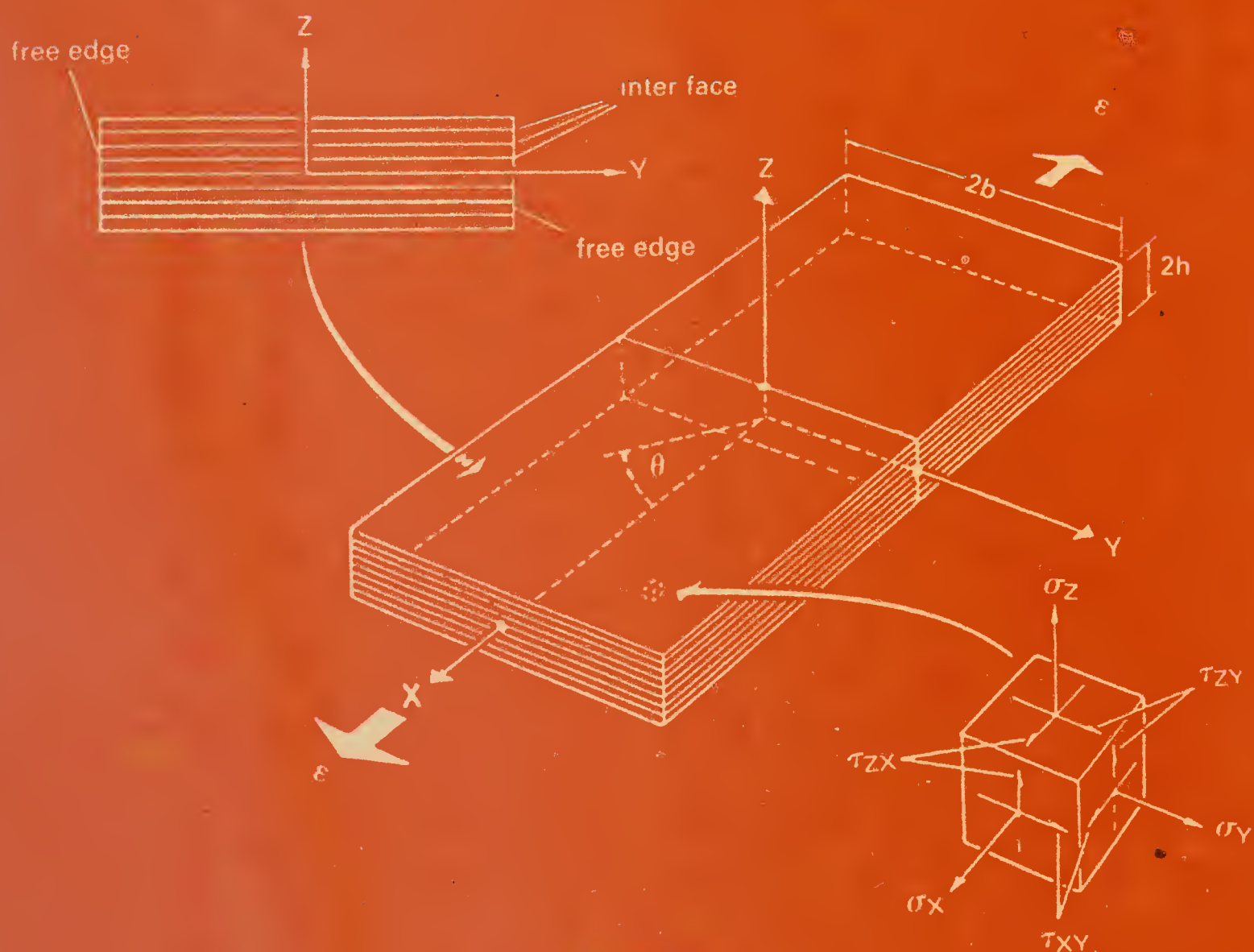


COMPOSITE MATERIALS AND STRUCTURES

Edited by

K A V PANDALAI

Indian Institute of Technology, Madras



INDIAN ACADEMY OF SCIENCES
Bangalore 560 080



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The cover shows the geometry and coordinate system of a unidirectional laminate,
from the paper by Soni & Pagano

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Reprinted from *Sāadhanā*—Academy Proceedings in Engineering Sciences
Volume 11, pp. 273–432, 1987

Edited by K A V Pandalai and phototypeset at Thomson Press, New Delhi,
for the Indian Academy of Sciences.

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Foreword

The breath-taking technological developments of the post-World War II period, be it in aerospace, microelectronics (particularly computers), nuclear engineering, fast communications or any other field, would not have been possible but for the development of appropriate new materials with the requisite characteristics and properties. Consequently, the area of Materials Science and Technology has become one of fundamental importance. Many countries have evolved national policies for R&D in Materials Science and these programmes are pursued with vigour. Thus, superconducting materials, gallium-arsenide for the development of superchips, fibre-optic materials, metal glass, aluminium-lithium and beryllium alloys, metal matrix and ceramic composites, high temperature polymer matrix composites, eutectic materials, and so on, are important areas of research and development at present. Composites are a class of materials that have revolutionized practically every sphere of industrial activity and technology during the last three decades or so. Although composites are of fundamental importance to technological progress in the area of aerospace, they, as also advanced ceramics and polymers, are used for a large variety of applications including those in the chemical, electrical, electronics, mechanical, transport and building industries, as well as defence-related applications. They also find applications in bio-medical engineering. High-temperature structural ceramics are estimated to bring about a fuel economy of around 20% in automobile engines. A market worth about 300 billion US dollars is predicted in the automobile industry alone. Admittedly, high-technology ceramics and advanced composites are high-cost materials and are primarily used for aerospace and defence-related industrial applications; but their large-scale use is bringing down the cost.

It is to focus attention on composite materials and their increasing engineering applications, particularly structural applications, that an International Conference on Composite Materials and Structures (ICCMS-88) was organized by the Fibre-Reinforced Plastics (FRP) Research Centre of the Indian Institute of Technology (IIT), Madras, in January 1988. In this Conference, about 50 papers were presented. But to focus special attention on certain specific issues and problems relevant to the field of composites, eleven 'invited papers' by some of the leading scientists in the field are included in this special issue of *Sādhanā*. All the other papers presented at the ICCMS-88 have been brought out as the Proceedings of the Conference, published by the Tata McGraw Hill Publishing Company, New Delhi.

In the first paper by Card & Starnes in this volume, an outline of the current research in composite structures at the Langley Research Center of NASA is presented. The authors discuss many of the important problem areas relating to composites that await satisfactory solution.

In the paper by Hayashi, who is one of the earliest researchers in the field of composites, the results of Japan's national R&D project on advanced composite materials, sponsored by the Agency of Industrial Science and Technology of the Ministry of International Trade and Industry of the Japanese Government, are presented. This paper also deals with some of the materials and processes developed in the area of plastic and metal matrix composites and presents the particulars of twelve products developed using composites.

The paper by Nair & Pandalai gives an outline of the R&D work in the area of composites at the FRP Research Centre of the IIT, Madras. The work reported relates to academic research aimed at generating scientific knowledge, R&D support provided to various high technology developments in the country and the development of certain FRP products that are useful and relevant to India. The paper also includes a brief outline of some of the on-going research studies at the FRP Research Centre.

The modelling of laminates for interlaminar stress estimation is discussed in the paper by Soni & Pagano. It reviews the contributions to the field by the authors and their co-workers over the last 15 years or so. One of the most promising theoretical models currently available is the local-global model developed by the authors, and an authoritative account of this model is presented.

Another paper on the theoretical modelling of laminated composite plates is presented by Krishna Murty who discusses the classical laminated plate and the higher order shear deformation models in order to indicate their interlaminar stress-predictive capability. The author also indicates some new possibilities for theoretical modelling of laminated composite plates based on his current research activity.

Sathyamoorthy focuses attention on the effects of geometric nonlinearity, transverse shear deformation and rotatory inertia on the vibration behaviour of single-layered plates and shells including laminated plates and shells made of filamentary composite materials. Recent developments in analytical methods of solution are emphasized.

Four papers by Shinohara and his co-workers deal with the development of eutectic metallic composites. Topics discussed are the eutectics of the binary, ternary and quaternary types made by directional solidification, measurement of the nine elastic constants of the Pb-Sn eutectic composites by pulse-echo techniques, preparation of certain eutectic composites, their microscopic examination and the experimental verification of their semiconductor properties, and, finally, in the fourth paper, the aluminium-copper alloys.

The paper by Kinra & Dayal reviews the nondestructive evaluation (NDE) techniques for composites focusing attention on ultrasonic testing and acoustic emission techniques. They also discuss the recently developed 'acousto-ultrasonic' technique, which combines acoustic emission with ultrasonics, and its application for characterization of fibre-reinforced composites.

Sincere thanks are due to all the authors of the articles published in this issue. The Indian Academy of Sciences, one of co-sponsors of the ICCMS-88, deserves special thanks for readily agreeing to bring out a Special Issue of *Sādhanā* on

composites containing all the invited papers. The help and cooperation of Professor R Narasimha, Chairman, Editorial Board of *Sāadhanā*, merits special mention. It is also a great pleasure for me to record the expert editorial help and cooperation given by Ms K Shashikala in bringing out this special issue.

K A V PANDALAI
Special Editor

Current research in composite structures at NASA's Langley Research Center

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Abstract. Research on the mechanics of composite structures at NASA's Langley Research Center is discussed. The advantages and limitations of special purpose and general purpose analysis tools used in research are reviewed. Future directions in computational structural mechanics are described to address analysis short-comings. Research results on the buckling and postbuckling of unstiffened and stiffened composite structures are presented. Recent investigations of the mechanics of failure in compression and shear are reviewed. Preliminary studies of the dynamic response of composite structures due to impacts encountered during crash-landings are presented. Needs for future research are discussed.

Keywords. Composite structures; computational structural mechanics; buckling of composite structures; crash-landings.

1. Introduction

Over the last ten years, graphite composite structures have been finding increasing applications in military and commercial aircraft. The types of applications occurring are a direct function of the designer's and decision maker's confidence in the underlying base technology for composite materials and structures. In the current state of the art, there is still a high risk associated with commitment to a heavily loaded primary composite structure for commercial aircraft.

One of the purposes of NASA's research programmes in composites is to reduce risk by obtaining fundamental understanding of the structural behaviour of composite structures, and with industry's help, to apply this understanding to the design of practical structures. A significant example of this approach can be found in the conduct of the NASA Aircraft Energy Efficiency (ACEE) programme. Researchers, designers and manufacturers worked together to develop flight-certified medium primary composite structures for commercial aircraft (Bohon *et al* 1983).

The purpose of the present paper is to review the lessons learned from past programmes and to relate these lessons to current research. The paper is limited to reviews of research in composite structures conducted at NASA's Langley Research Center. In the paper, the results of the ACEE programme are discussed first. Then a

review of the analysis tools used in research on composite structures is presented. Future directions in computational structural mechanics research to alleviate analysis shortcomings are described.

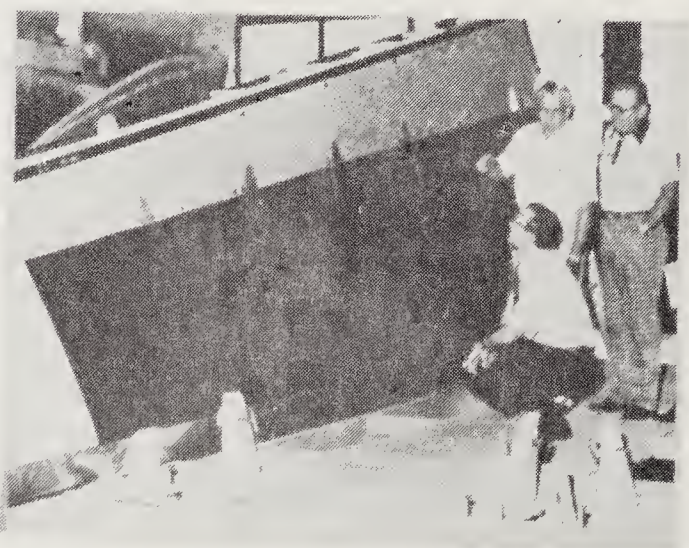
Experimental and analytical results on the buckling and postbuckling behaviour of composite panels are presented. Research results on the mechanics of compression and shear failures are then discussed. Finally, results of preliminary research studies on the dynamic response of composite structures to crash-type loadings are reviewed.

2. ACEE component failures

The medium primary structural components investigated in the NASA ACEE programme are pictured in figure 1. They consist of large horizontal and vertical stabilizers, and a large vertical fin. The components are all primary structures in that failure of the component would result in loss of the aircraft. They have been described as medium primary structures since they are empennage structures which do not experience the severity of loading applied to a fuselage or wing structure. Nevertheless,



L-1011 (9 x 25 ft) vertical fin



737 composite horizontal stabilizer (7 x 23 ft)



DC-10 composite vertical stabilizer (4 x 17 ft)

Figure 1. ACEE medium primary structural components.

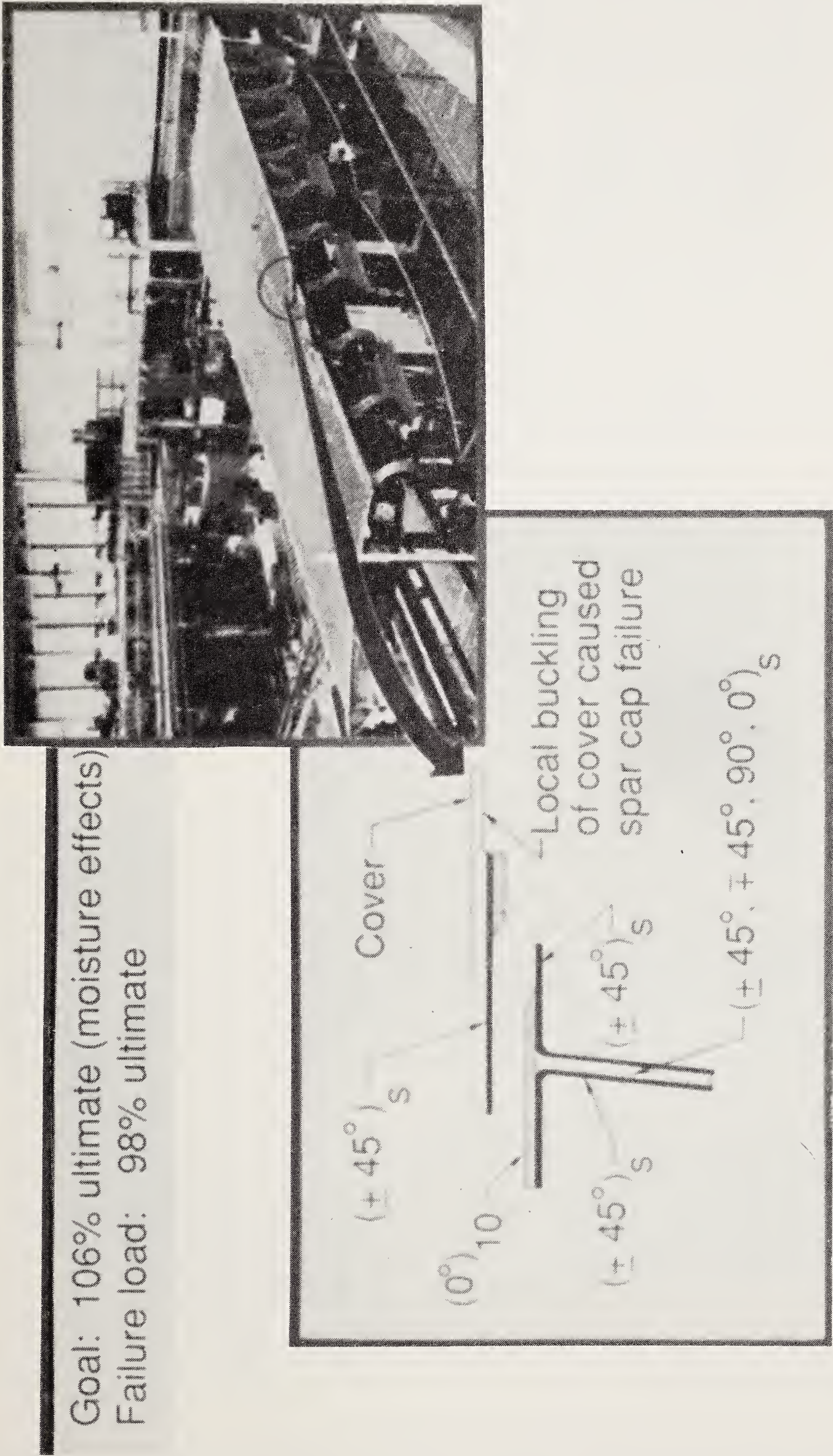


Figure 2. Failure model of L-1011 vertical fin.

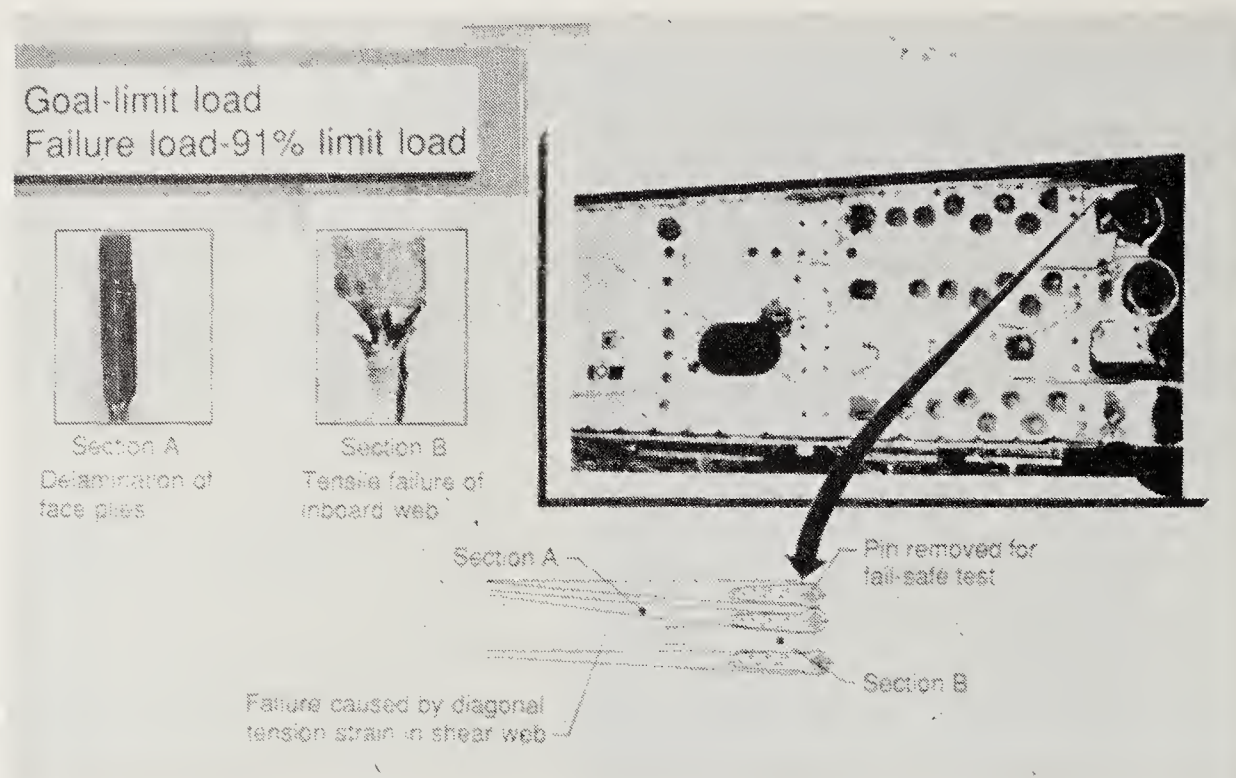


Figure 3. Failure mode of 737 horizontal fin.

the design of these graphite-epoxy structures proved to be a significant challenge.

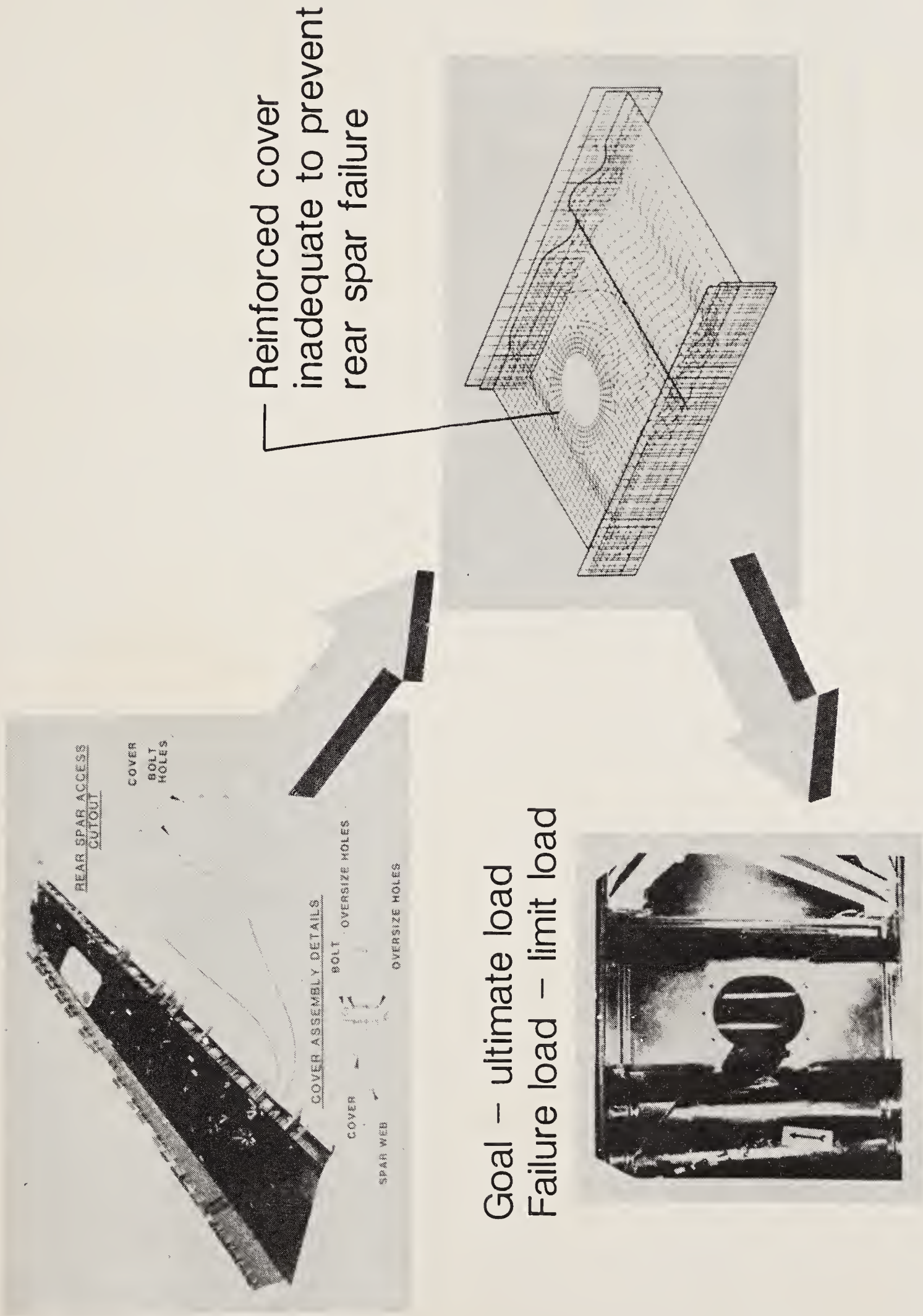
As part of the ACEE programme, each of the components was to undergo FAA certification for possible use in commercial aircraft. This process required extensive analyses and ground testing culminating in full-scale static and fatigue tests. In each of the component static tests a premature failure was experienced near limit or ultimate load. The type of failure and loading condition for each component is presented in figures 2, 3 and 4. In each case, failure was precipitated by an unexpected local design error which produced a catastrophic global failure in the component.

For the L-1011 vertical fin (figure 2), local buckling of the stabilizer box skin cover caused local failure in the spar cap. It was found that repeated buckling under cyclic loads could introduce undesirable interlaminar tensile stresses into the cap laminate. In the 737 horizontal fin (figure 3), redistribution of load with a lug pin removed for fail-safe testing resulted in high tensile strains precipitating a failure of the shear web between lugs. Subsequent investigation revealed that the web buckled in shear at 50% of limit load producing very large tension field strains in the web at failure. For the DC-10 vertical stabilizer (figure 4), a bolted cutout reinforcement cover plate was found to be ineffective in carrying the required web design loads and resulted in failure of the rear spar of the component.

Failures attributed to local detail design errors are not uncommon in static and fatigue tests of metal aircraft structures. Unfortunately for composite structures, the resulting redistribution of loading due to the error sometimes results in more catastrophic failures of critical elements. The lessons learned from the ACEE component tests are that composite designers must develop better understanding of local behaviour, and need better design and analysis tools to do their job.

3. Research analysis tools

A histogram of analysis tools developed in research programmes is presented in figure 5. The tools are distinguished by being either special purpose or general purpose analyses. Table 1 contains the source and attributes of each of the computer codes. While special



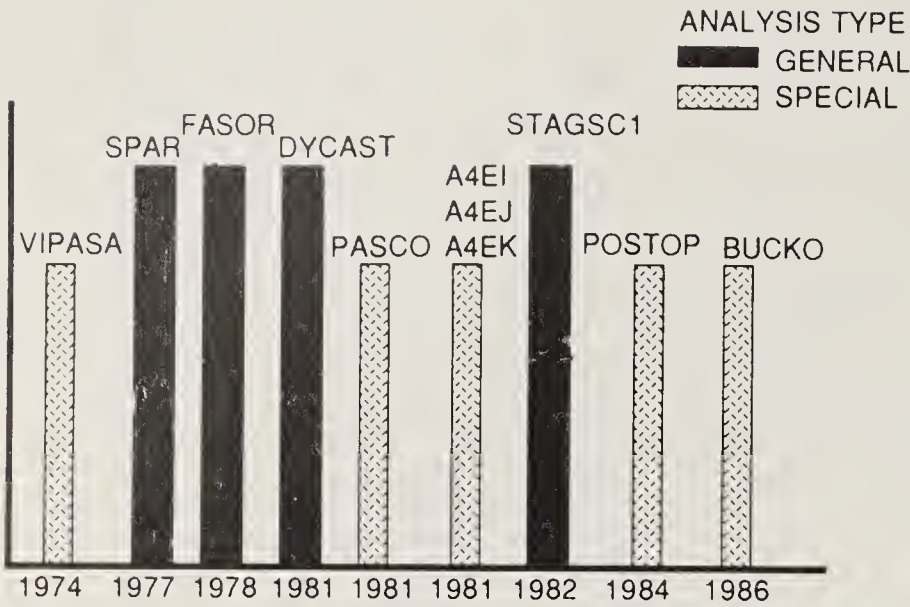


Figure 5. LaRC research analysis tools.

purpose codes are very restricted in their application, they are invaluable in preliminary design. An example of the utility of a special purpose analysis (BUCKO, Nemeth *et al* 1986) is presented in figure 6. The analysis uses trigonometric series and an energy minimization approach to find approximate solutions for buckling of a plate with a circular cutout. Sample buckling results are presented on the left of figure 6 in a plot of buckling coefficient versus cutout diameter to plate width for square plates. The plot shows the unexpected results that the strength of the plate increases with cutout size for $\pm 60^\circ$ laminates. The trend has been confirmed by the experimental results shown on the right of the chart and occurs because of redistribution of stresses around the cutout (see Nemeth 1986). Using this tool, extensive parametric studies have been performed on the effects of plate width and various plate boundary conditions.

Table 1. Structural analysis computer codes.

Analysis tool	Features	References
VIPASA	Vibration and buckling of stiffened composite	Wittrick & Williams (1974)
SPAR	Computationally efficient finite element analysis code	Whetstone (1977)
FASOR	General shell of revolution analysis for composites including transverse shear	Cohen (1986)
DYCAST	Large deflection, nonlinear analysis for impact loading	Pifko & Winter (1981)
PASCO	Panel sizing analysis for buckling of stiffened composite panels	Stroud & Anderson (1981)
A4EI A4EJ A4EK	Stiffened composite bonded and bolted joint analysis	Hart-Smith (1981)
STAGSC-1	General purpose nonlinear finite element analysis	Almroth <i>et al</i> (1982)
POSTOP	Sizing analysis for postbuckling of composite panels	Dickson & Biggers (1984)
BUCKO	Approximate analysis of buckling of composite panels with cutouts	Nemeth <i>et al</i> (1986)

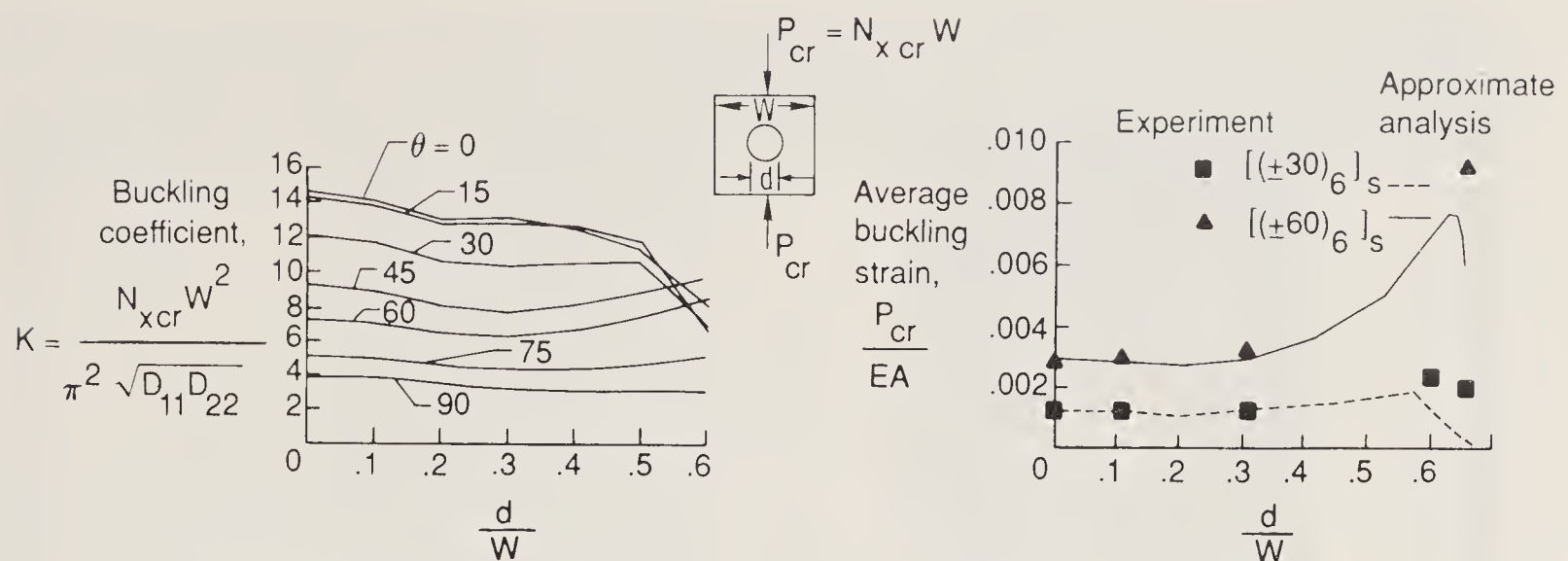


Figure 6. Results from special purpose analysis of compressive buckling of plate with cutout.

4. Computational structural mechanics

Future projections of analysis needs for complex structures have resulted in NASA sponsorship of a new programme in computational structural mechanics (CSM) at the Langley and Lewis Research Centers. The programme focuses on new analysis techniques to exploit the computing capability of the 90's. Activities at Langley are summarized in figure 7 taken from Knight & Stroud (1985). The programme addresses problems in static and dynamic analyses of aircraft and spacecraft structures. Efforts are being conducted to learn how to exploit the capabilities of supercomputers such as the Cray 2 shown in the figure. A generic test bed has been established at integrate the methods developed by researchers in universities and industry.

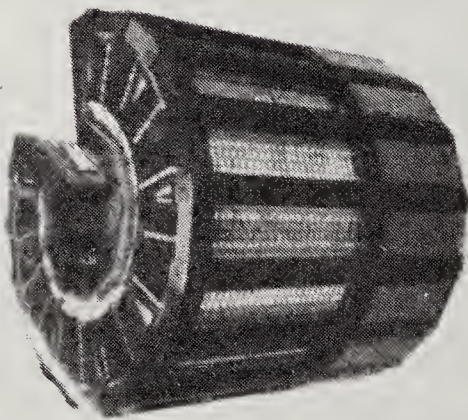
A research thrust for composite structures is the development of local three-dimensional nonlinear analysis within a larger two-dimensional analysis model. Such a capability would address complex local design problems and permit through-the-thickness failure criteria to be applied. In the Langley CSM activities, the graphite-epoxy stiffened panel shown at the left of figure 7 is being used as a focus problem to help identify analysis needs. The interrupted stiffener with cutout produces a difficult-to-predict nonlinear global-local response. Test results exist for the panel which will be used to verify new analysis techniques.

A key computational element in making such computations more routine is the exploitation of parallel processing. Langley is actively working this area using a FLEX/32 multiple-instruction multiple-data computer with both local and shared memory. The capability of this computer is presented in figure 8. A vibration solution for the composite stiffened panel focus problem (Bostic & Fulton 1987) was recently obtained and is presented in figure 9. The results for extraction times for the first five eigenvalues of the clamped stiffened plate using the Lanczos method indicates an actual computational speedup of a factor of about 12 using 16 FLEX processors, when all of the overhead computational time is included in the comparison with the theoretical or potential speedup. While the vibration results illustrate improvements only in two-dimensional aspects of the focus problem, the potential of parallel processing for speeding up the analysis computations is apparent.

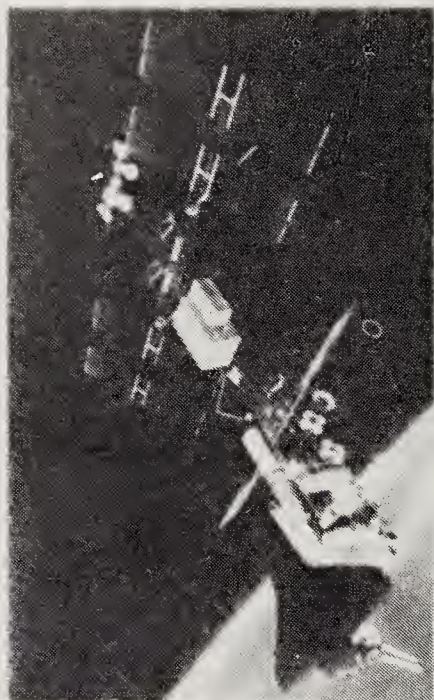
Aircraft Structures



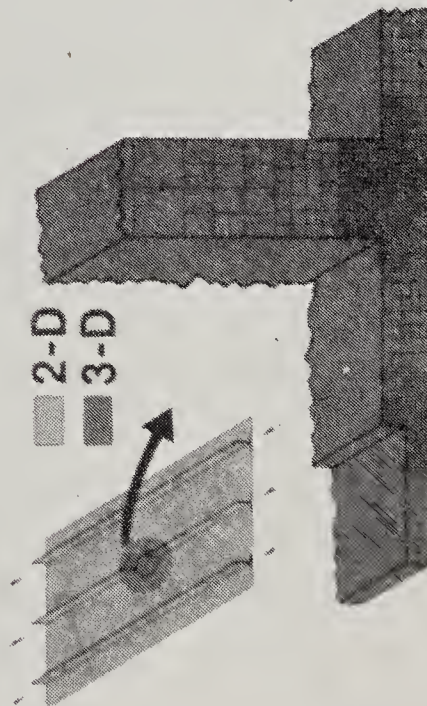
Advanced Architecture
Computers



Space Structures



Local 3-D nonlinear stress analysis
within larger 2-D analysis model



Transient Dynamics

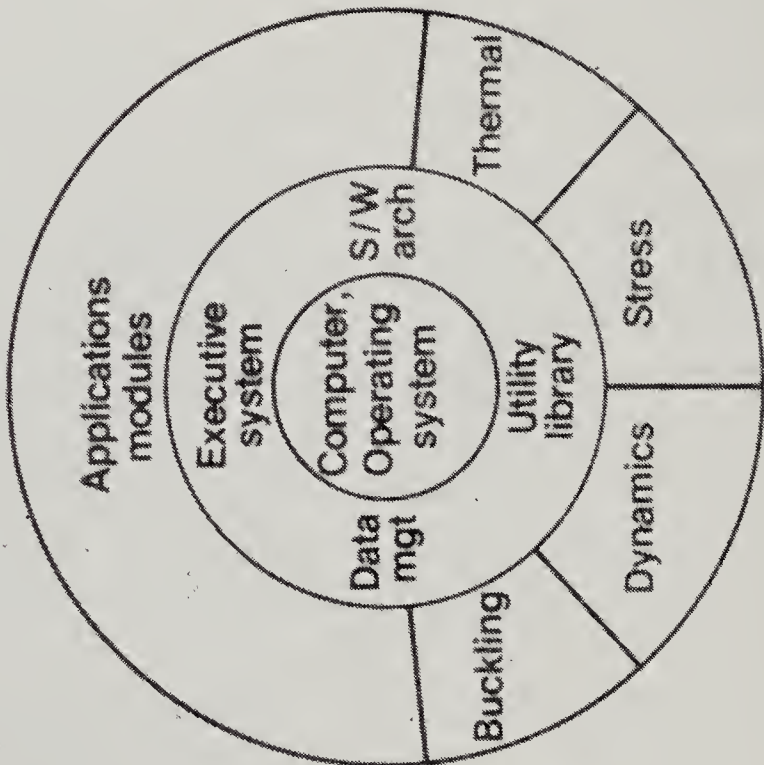
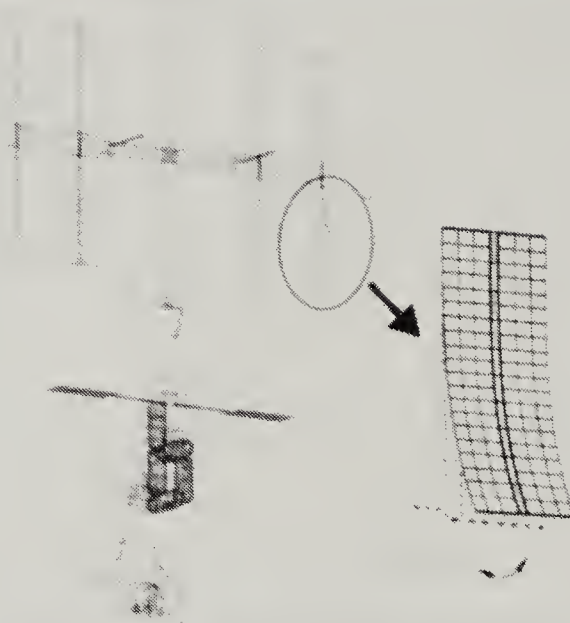


Figure 7. Computational structural mechanics.

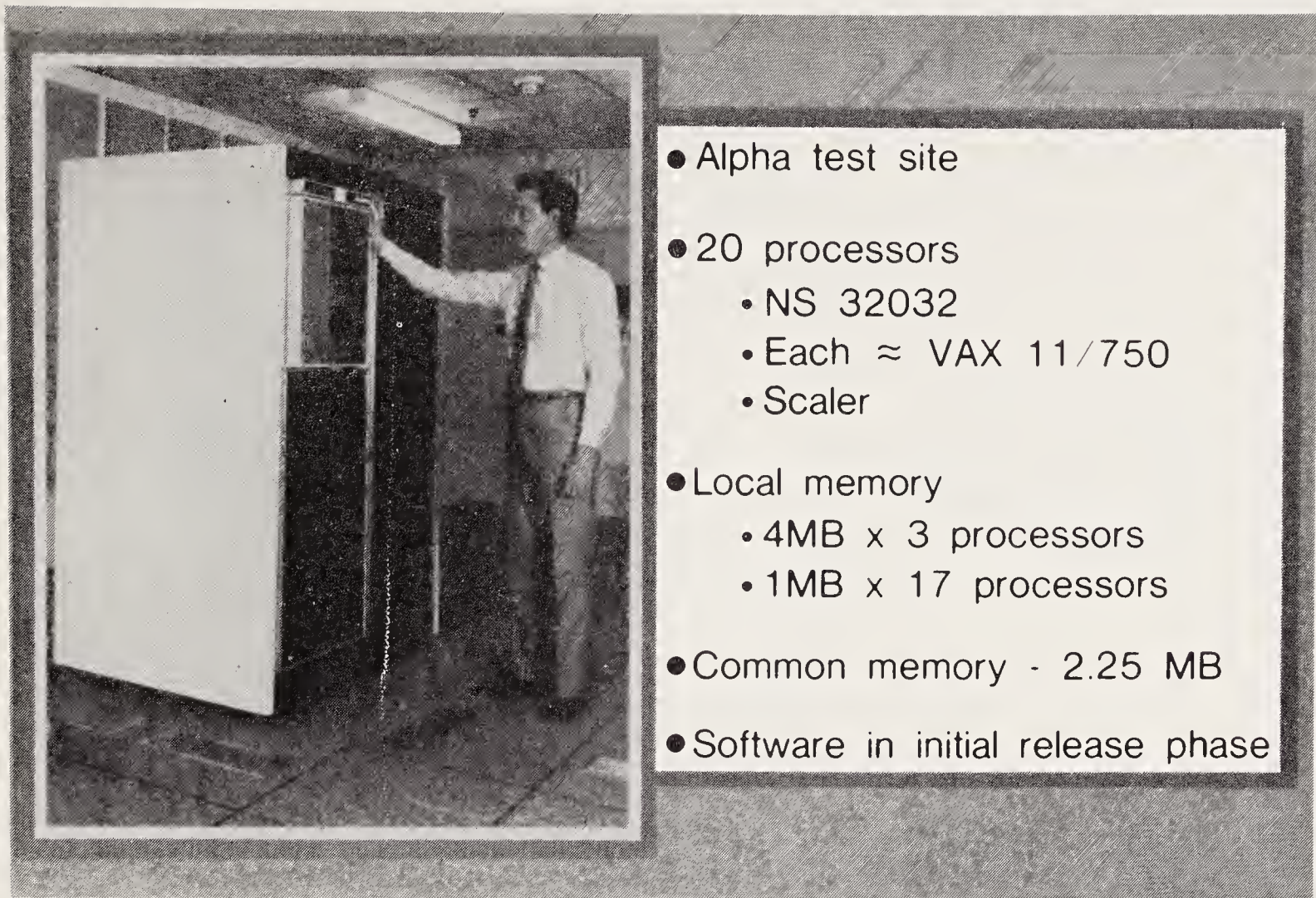


Figure 8. FLEX/32 multiprocessor computer.

5. Buckling/postbuckling theory

5.1 Buckling

The state of the art in predicting buckling of stiffened and unstiffened composite plates is relatively mature. Special purpose codes exist which are computationally efficient and embody all of the classical results for metallic plates. An example of such an analysis is presented in Williams & Anderson (1985). The major difficulty in this type of analysis has been the obtaining of accurate solutions for combined in-plane shear and compression loadings. The method of Lagrangian multipliers has been employed to

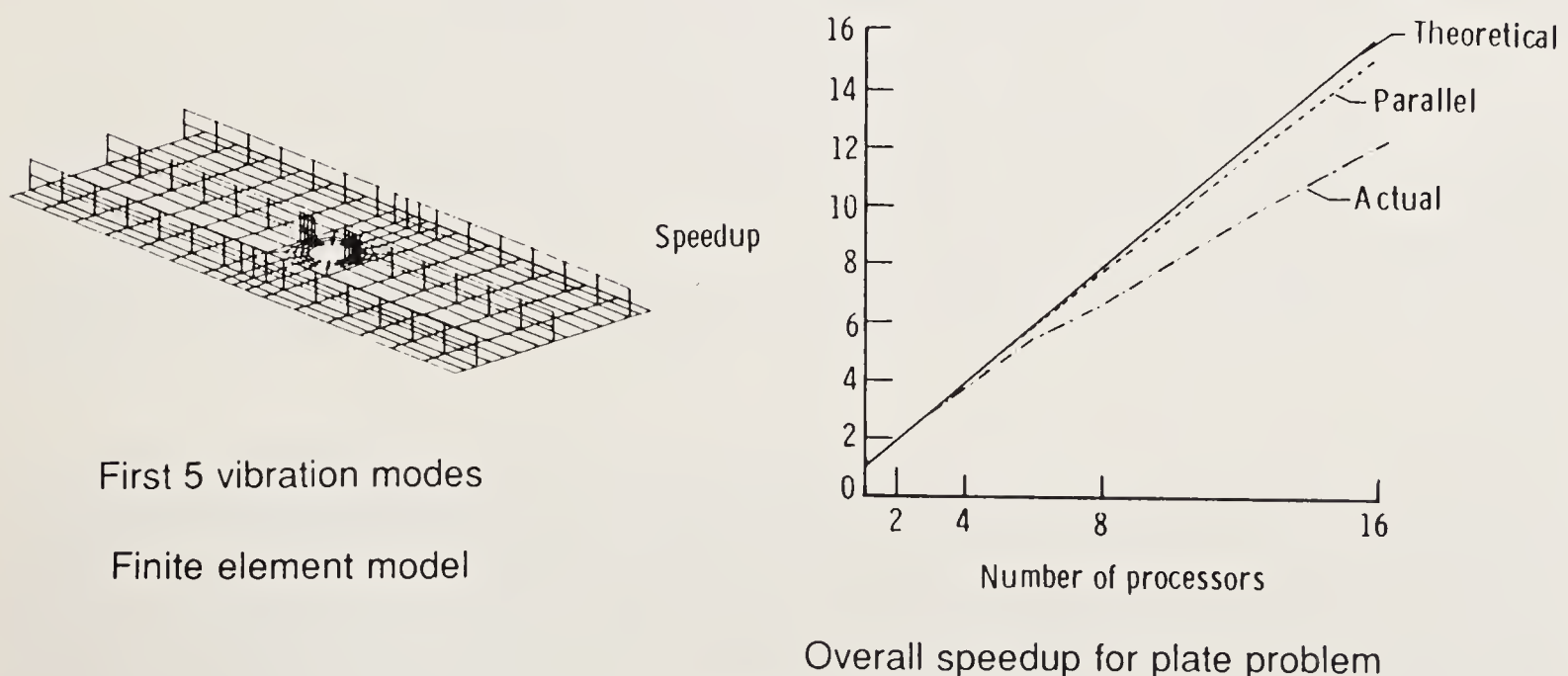


Figure 9. Parallel computation results.

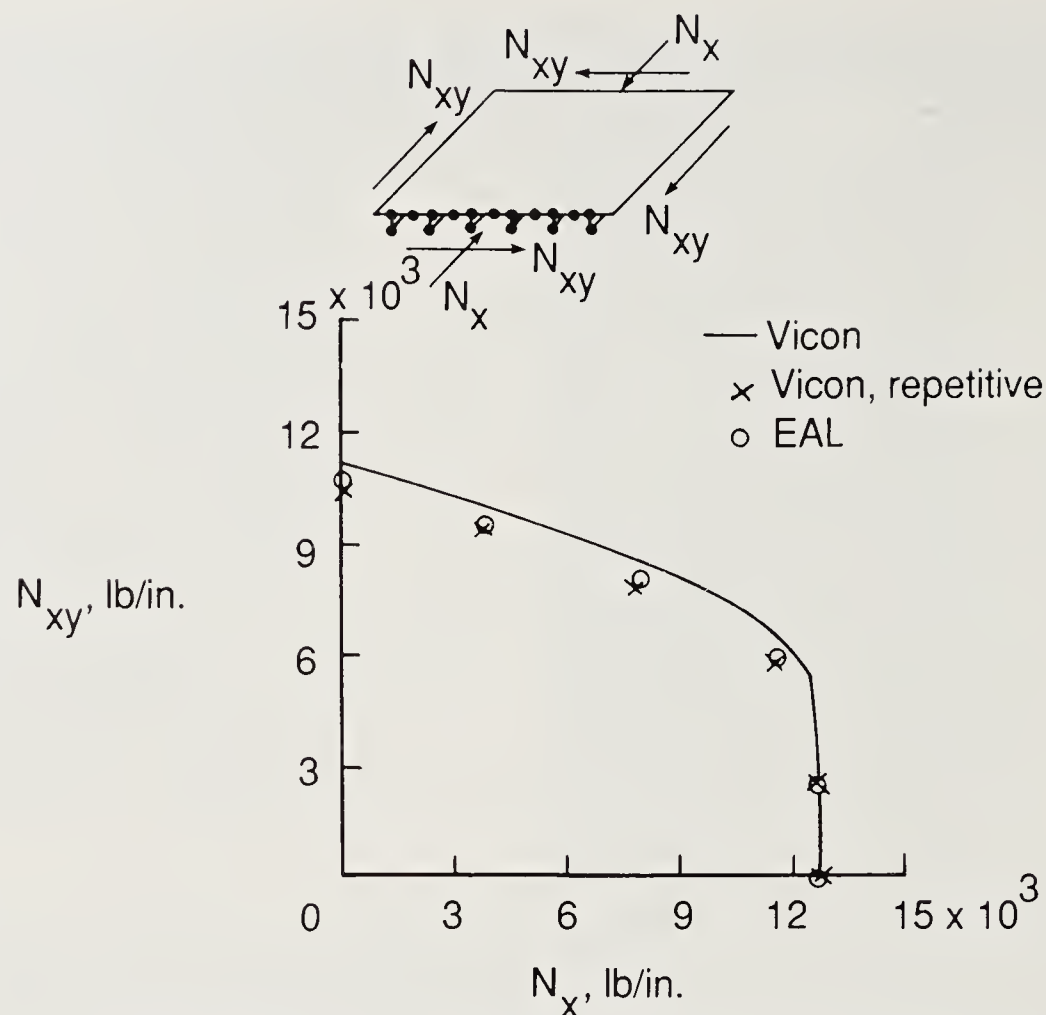


Figure 10. Shear/compression buckling interaction curve.

provide proper constraints for structures with elastic or rigid supports.

An application of this technique (Williams & Anderson 1985, pp. 51–71) is presented in figure 10. Buckling interaction curves for combined shear and compression loading of a simply supported stiffened graphite-epoxy panel are shown. A comparison of buckling results using finite element analysis (EAL) shows excellent agreement with the more computationally efficient special purpose analysis results (VICON).

5.2 Postbuckling

The state of the art in postbuckling analysis is less mature. Classical analytical techniques have been applied (Stein 1985, pp. 205–223) to estimate the postbuckling stiffness of long plates subjected to either compression or shear. Typical results for simply supported and clamped composites plates are presented in figure 11. For the $\pm 45^\circ$ graphite-epoxy plate shown, the compressive buckling stiffness is about half that of the unbuckled plate. In the case of shear, the stiffness has only a small reduction for the $v = 0$ boundary condition and is about half the stiffness for the $N_y = 0$ boundary condition. More approximate analyses using the approximate “effective width” formulations developed by Koiter and van der Neut have been developed for engineering analysis and design in Dickson & Biggers (1984). Much work remains, however, to develop and verify practical engineering estimates of postbuckling stiffness.

Progress has been made in more complex analysis of postbuckling of plate and shell structures. A difficult problem is the finite element analysis of structures in which multiple bifurcation points exist in the postbuckling regime. In Thurston *et al* (1985) a change of variables is introduced in the problem which is computationally efficient near bifurcation points. Results presented in figure 12 for postbuckling of an orthotropic plate loaded by in-plane compression were obtained with a significant decrease in

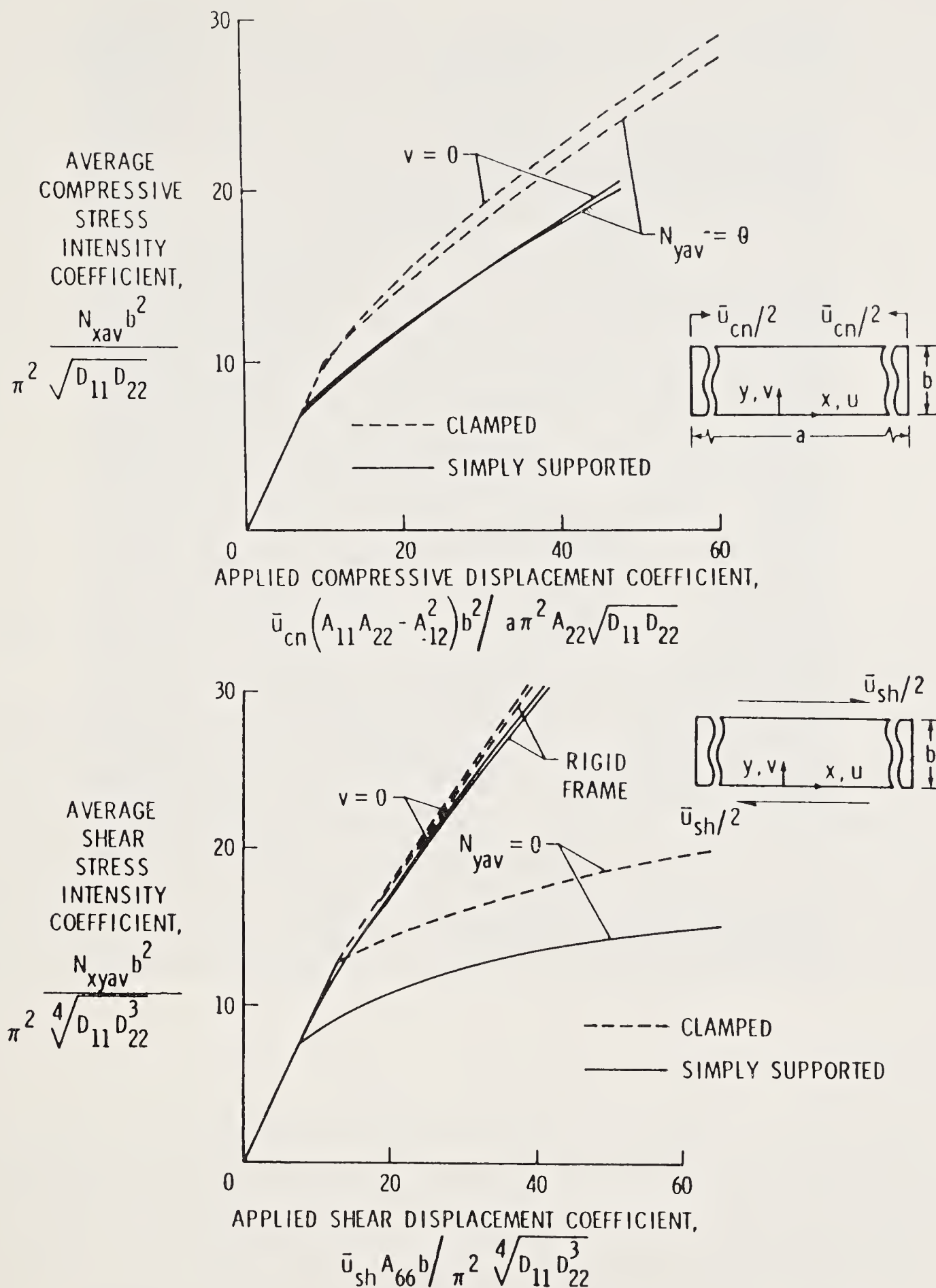


Figure 11. Postbuckling of long plates under compression and shear loadings.

computational effort for this method (squares on figure) over conventional Newton techniques (circles on figure).

5.3 Correlation with experiment

Applications of general purpose finite element analysis to postbuckling of composite plate structures have been remarkably successful. Rouse (1985) presents studies of shear postbuckling in graphite epoxy plates. Typical results are presented in figure 13 for a quasi-isotropic specimen. The shear strains derived from back-to-back strain gauge rosettes show good agreement with analysis up to 2% strain. Similar agreement has

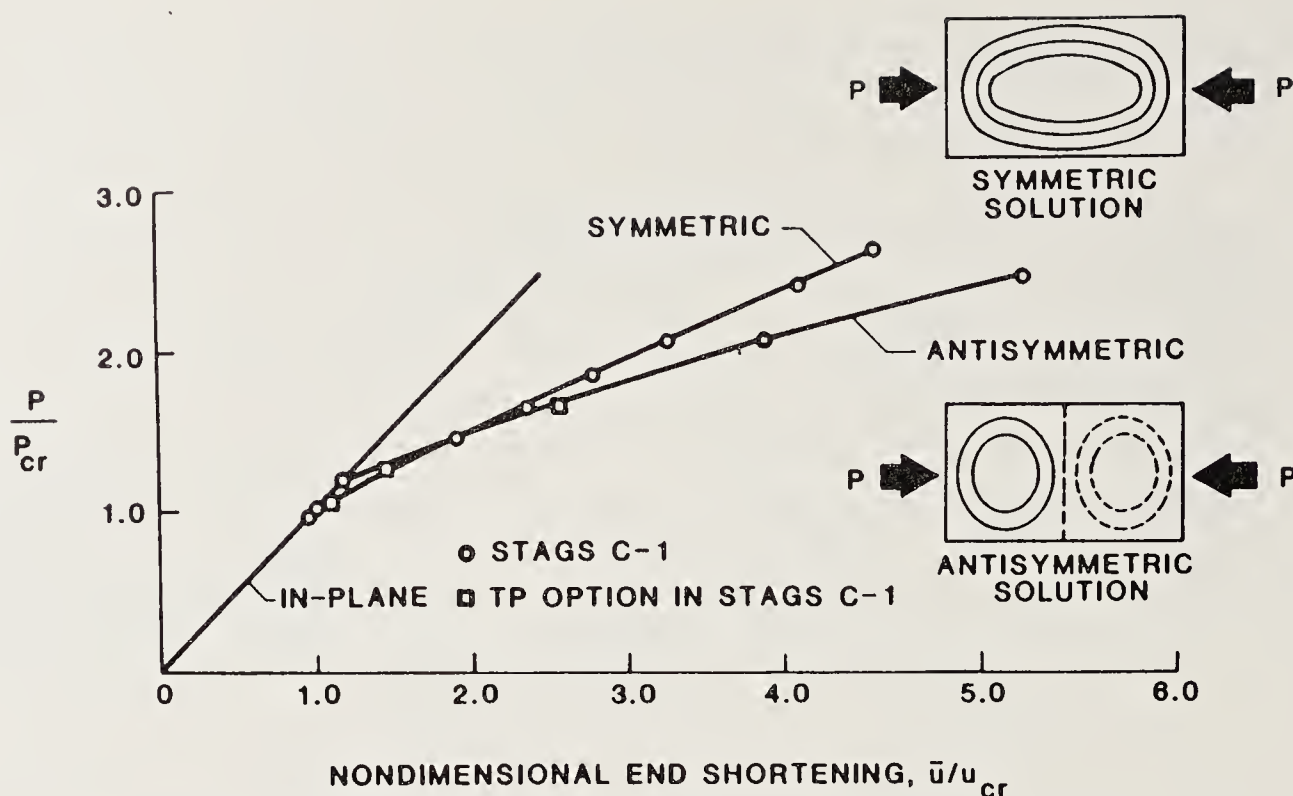


Figure 12. Postbuckling of a compression-loaded orthotropic plate.

been obtained by Starnes *et al* (1985) for flat stiffened composite panels loaded in compression and typical results are shown in figure 14.

Postbuckling analysis of curved panels have proven to be more difficult. In Knight *et al* (1986) the STAGSC-1 Almroth *et al* (1982) shell analysis has been applied to predict the postbuckling behaviour of ten graphite-epoxy curved panels. The analysis includes the effects of initial geometric imperfections, predictions of the characteristic “jump” to stable postbuckling behaviour and estimates of first-ply failure in the graphite-epoxy laminate. Typical load-shortening behaviour is illustrated in figure 15. Results shown indicate good correlation up to buckling with deteriorating agreement thereafter. The most challenging problem in postbuckling is the accurate prediction of ultimate failure.

6. Compression/shear failure

Theoretical research, together with carefully conducted experiments, is revealing some of the mysteries of failure of composite structures. A long-standing problem has been the prediction of the ultimate compression strength of composite laminates. Experiments (Starnes & Williams 1983, pp. 283–306; Stuart & Williams 1986) have revealed the existence of two modes of failure usually not considered in composite strength considerations.

The first mode is transverse shear crippling (figure 16). This mode of failure is well-known in sandwich structures, but rarely observed in composite laminates. Usually, compression-loaded composite materials will fail by delamination. However, recent improvements in composite matrix materials have suppressed delamination so that the ultimate strength is determined by shear crippling.

The existence of delamination and shear crippling requires development of accurate theories accounting for through-the-thickness stresses in composite laminates. In Stein (1985) a transverse shearing theory has been developed using a trigonometric expansion of displacements in the thickness direction. Typical results for buckling of thick aluminum plates (Stein & Bains 1986) are presented in figure 17. The results suggest that significant buckling strength reductions can occur in very thick metal and

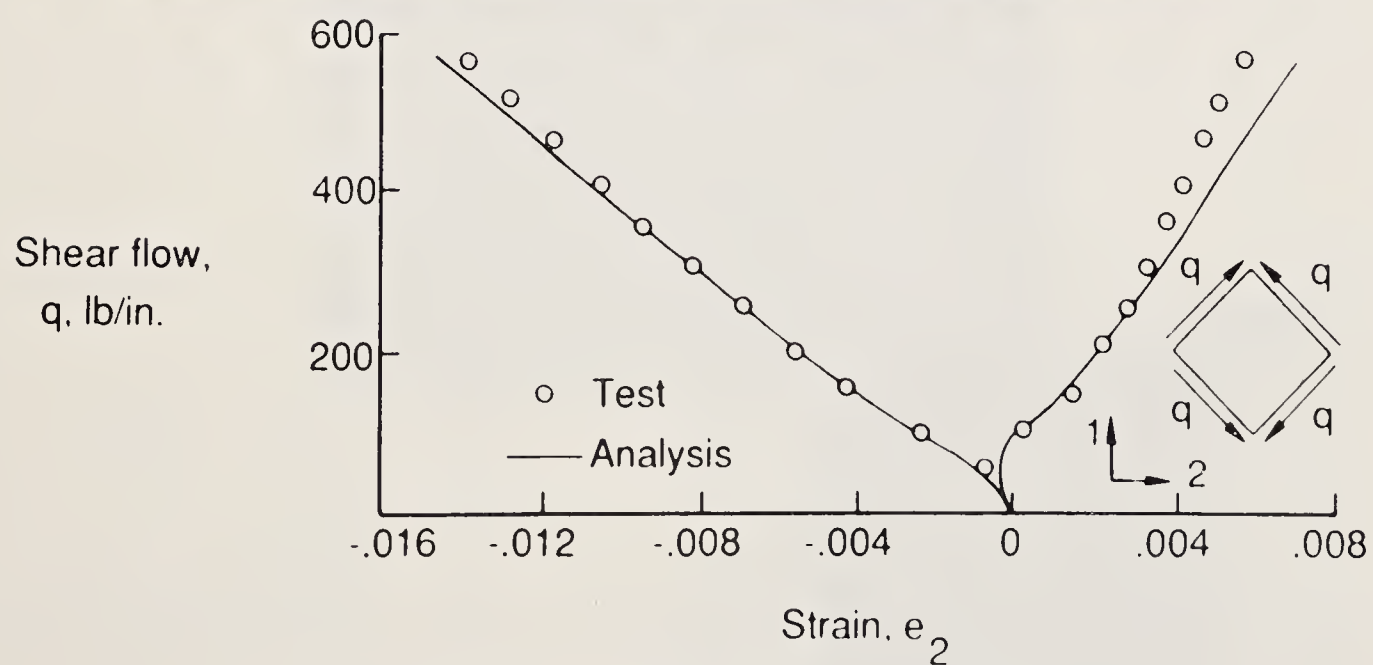
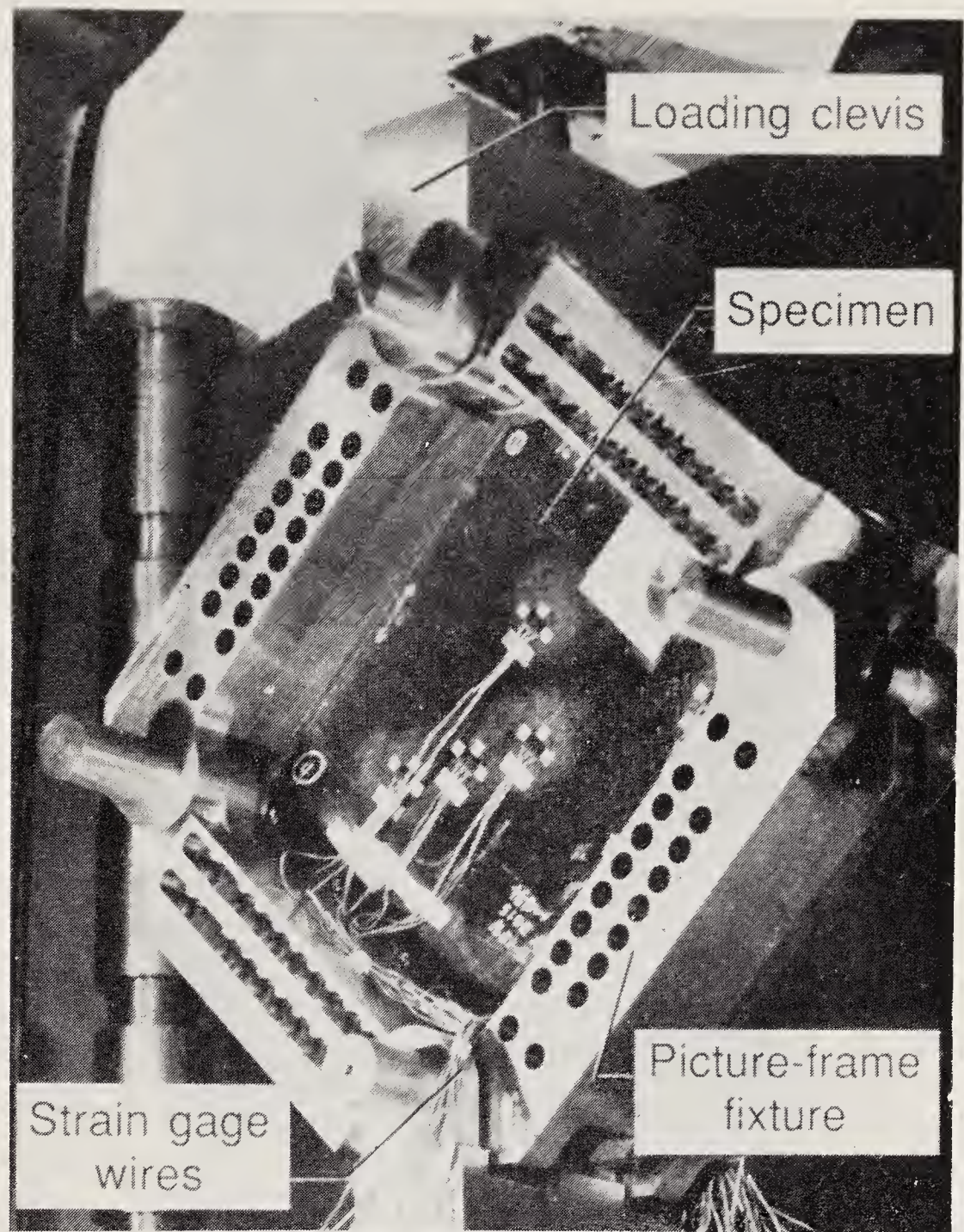
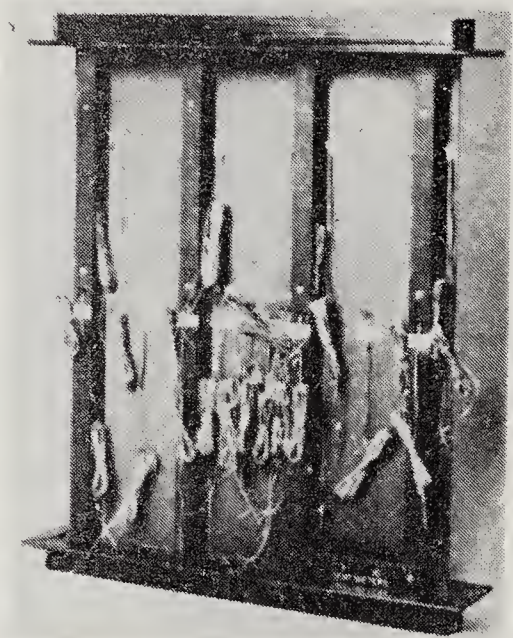


Figure 13. Shear postbuckling of graphite-epoxy plate.

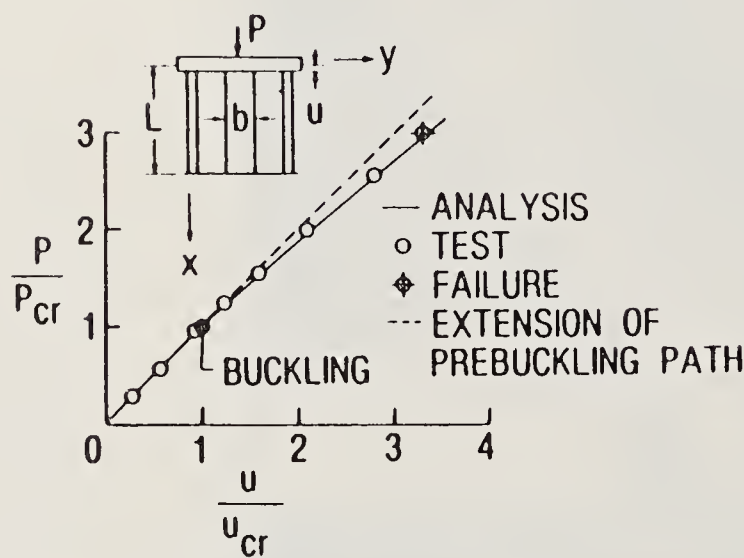
composite structures because of transverse shear flexibility. In related work (Reddy & Liu 1987), an accurate transverse shear theory was developed using up to cubic terms in the trigonometric expansion. This result permits the application of the theory to finite element analysis.

A second observed mode of failure in composite laminates is in-plane matrix shearing (Shuart & Williams 1986). Figure 18 presents compression failure results for a $\pm 45^\circ$

TYPICAL GRAPHITE-EPOXY TEST SPECIMEN



END SHORTENING



SURFACE STRAINS

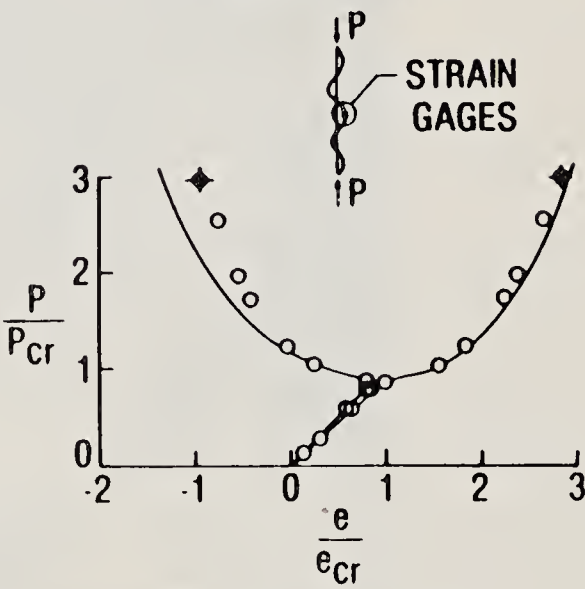


Figure 14. Compression postbuckling of a flat stiffened graphite-epoxy panel.

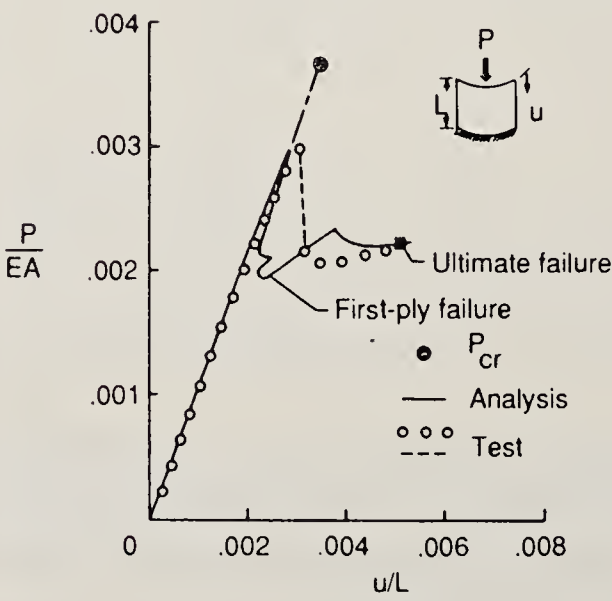
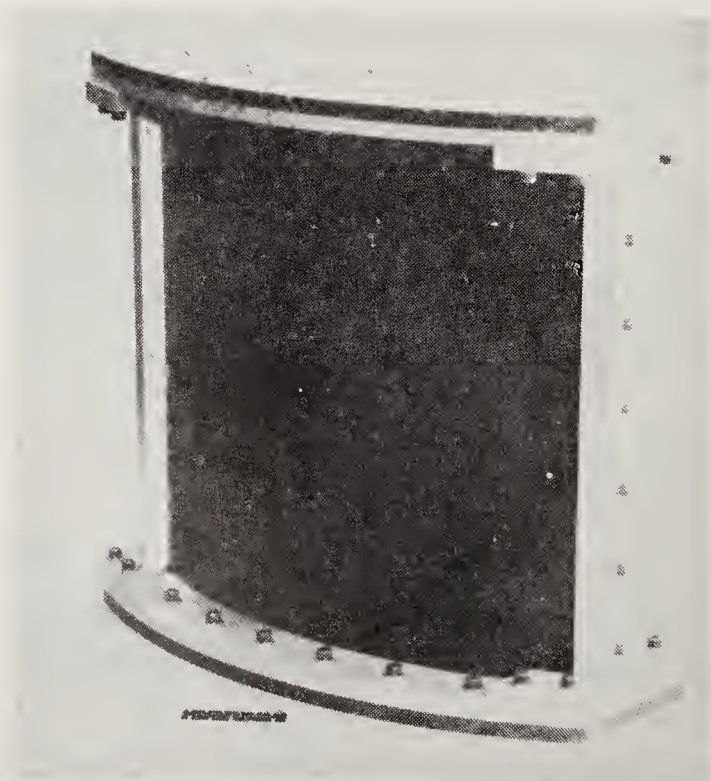


Figure 15. Compression postbuckling of curved graphite-epoxy plate.

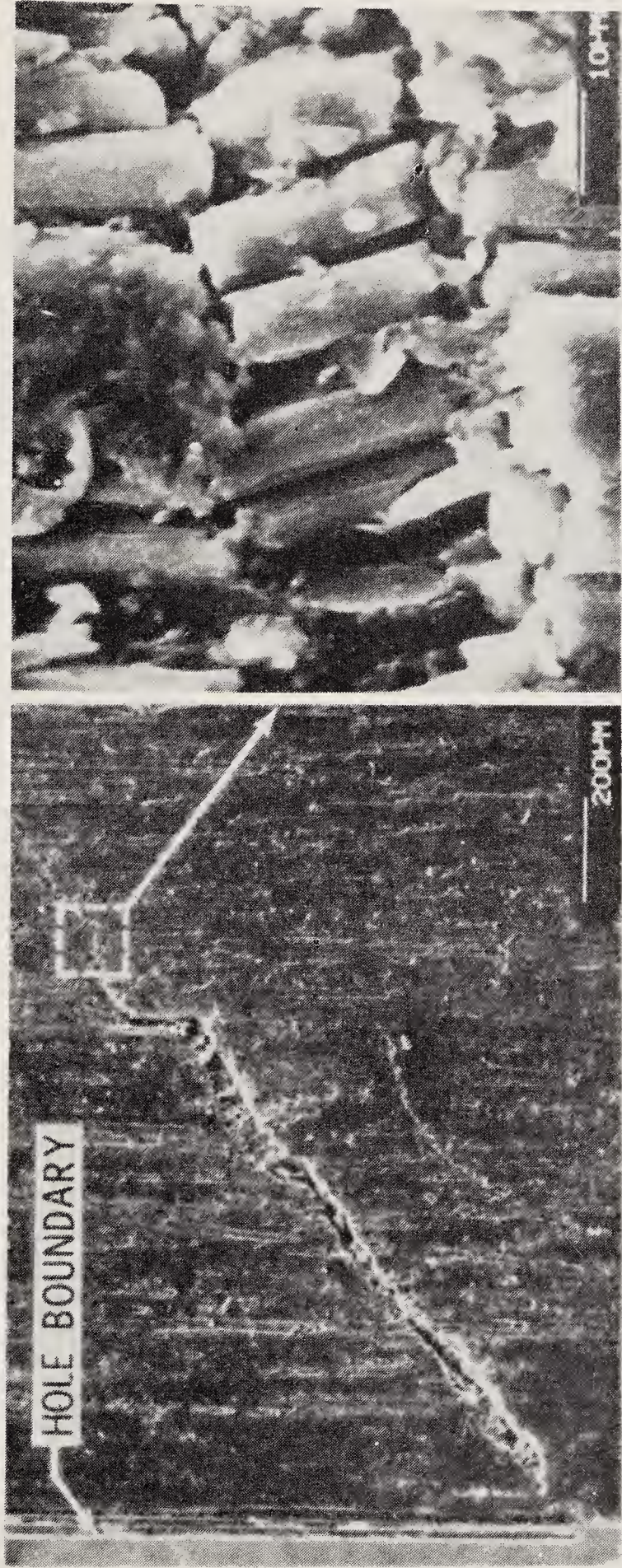
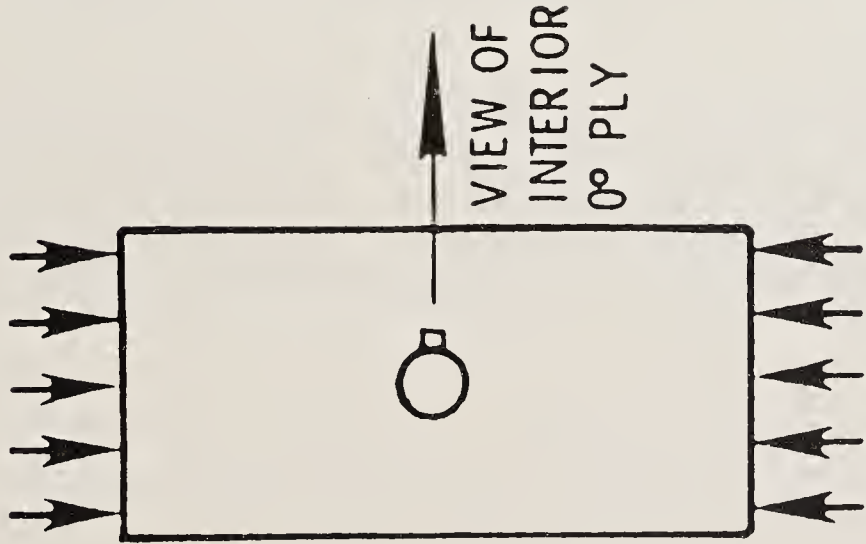


Figure 16. Transverse shear failure mode.

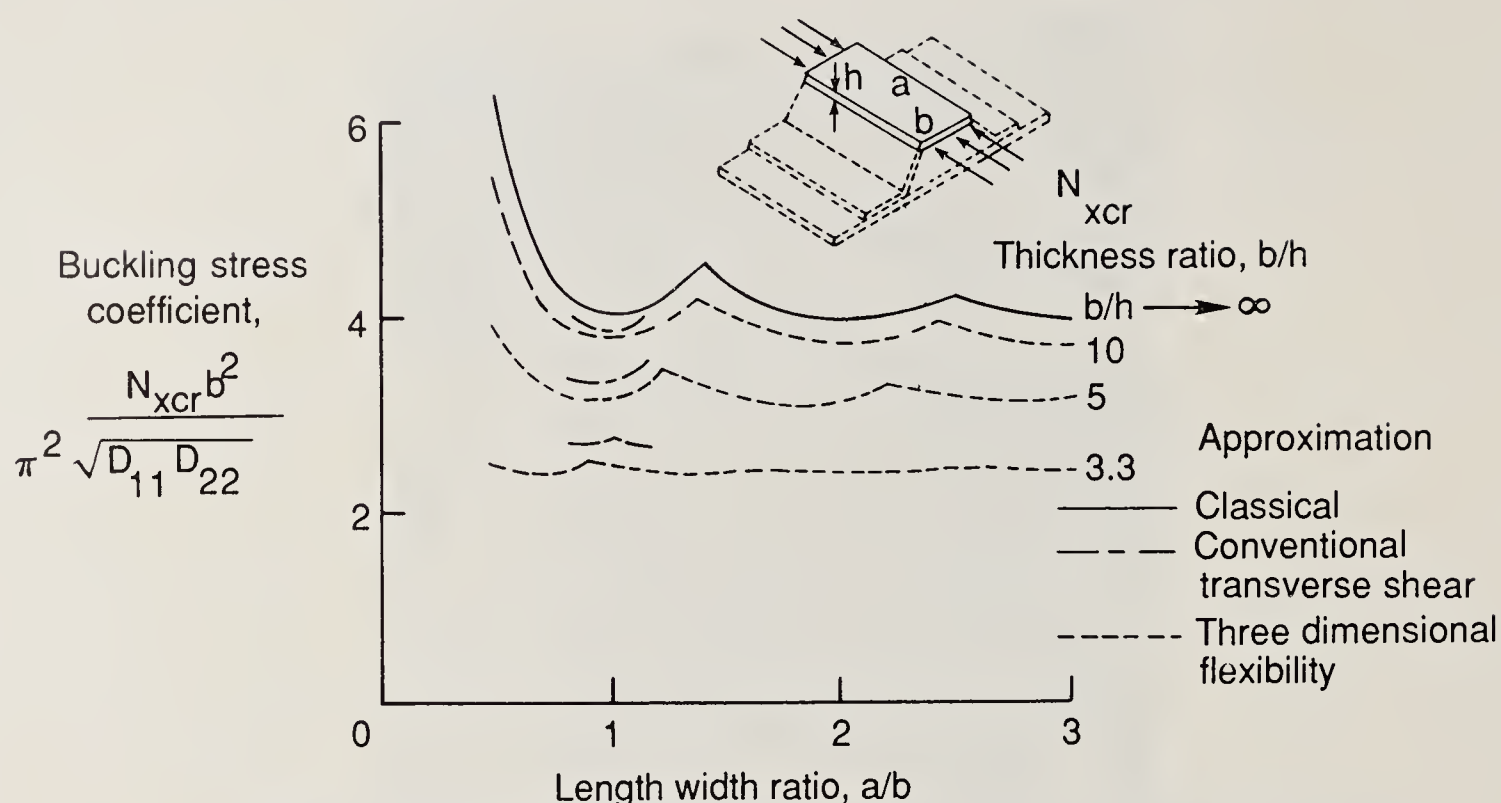


Figure 17. Transverse shear effects on buckling of aluminum plates.

laminate. Studies of failed specimens using birefringent material or C-scan techniques revealed the existence of in-plane matrix shearing bands as seen in the figure. The failure mode must be accounted for in laminates with plies oriented at or near 45° .

A general theory of failure based on microbuckling in laminates has been developed by Shuart (1985). As suggested in figure 19, the theory treats each lamina as a fibre layer supported on an elastic foundation of matrix material. Solutions for buckling are obtained using short-wavelength approximations. Results shown on the figure for angle-ply laminates indicate that interlaminar shear failures occur in laminates with ply orientations from $0-20^\circ$. In-plane shear failures occur for ply orientations from $20-70^\circ$. While the basic strength trends seem to be predicted by this theory, efforts are continuing to improve the correlation between theory and experiment.

7. Dynamic response

The application of composite materials to primary structures in aircraft suggests that consideration be given to the crash-worthiness of composite aircraft structures. A research programme to address some of the basic issues in dynamic response of composites has been initiated at NASA Langley (Carden 1985).

Friction and wear of composite fuselage skins can be an important consideration especially in the event of an emergency sliding (belly) landing on a runway surface. Some fundamental data on friction and wear of conventional composite materials are presented in figure 20 taken from Jackson (1985). The data were generated by dragging composite and metallic specimens over a standard runway surface at various speeds. Friction results show that graphite coupons and panels have roughly half the coefficient of friction of metallic specimens, so that composite aircraft would be expected to have a longer slideout. The wear data presented show that wear rates for composite specimens were from two to five times larger than metallic specimens.

Basic research on the dynamic response of fuselages to impacts encountered during crash-landings has also been initiated. The characteristic shape of a metallic fuselage after a vertical drop test (Williams & Hayduk 1983) is shown in figure 21. Much of the

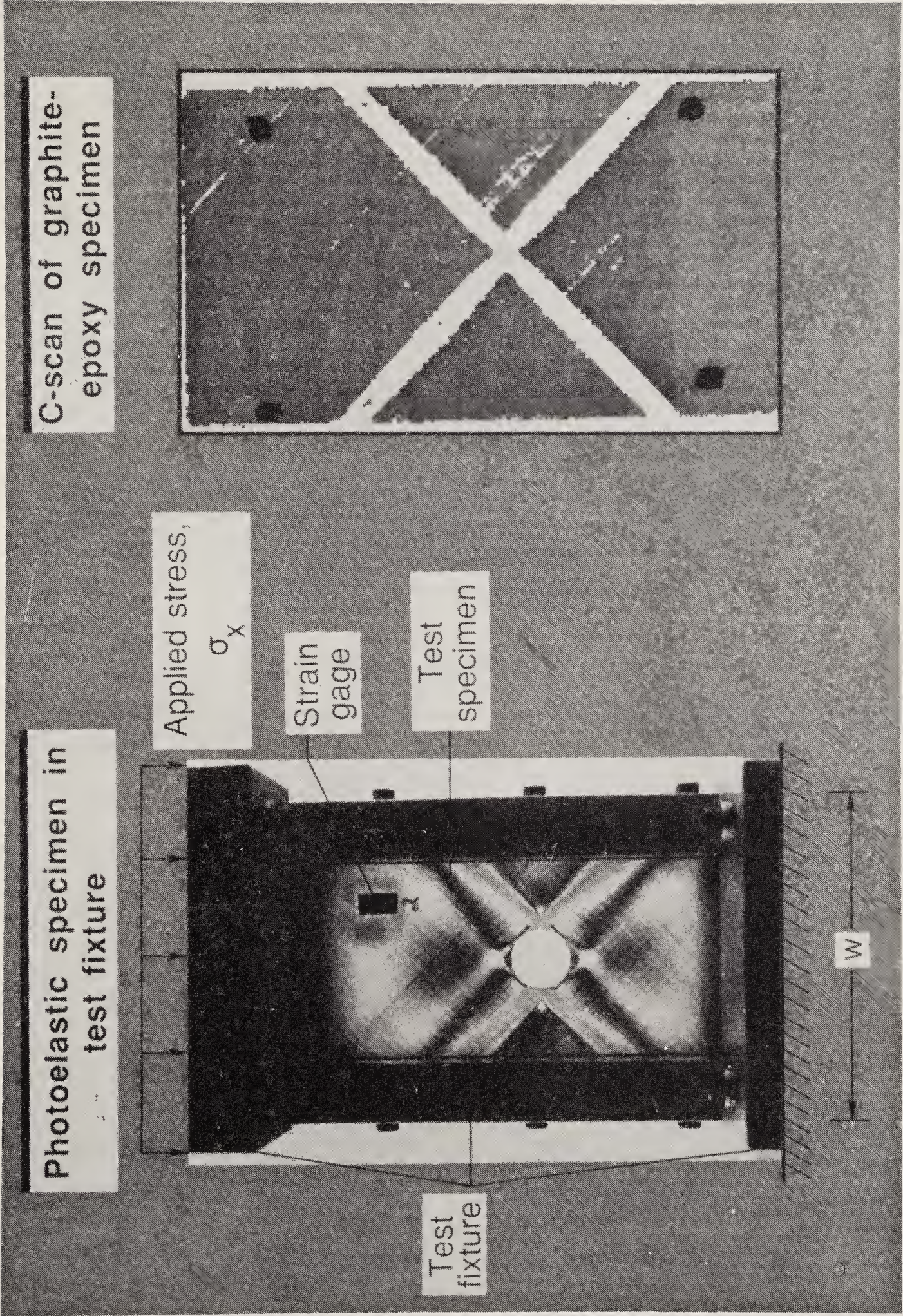


Figure 18. In-plane shearing failure in graphite-epoxy plates with holes.

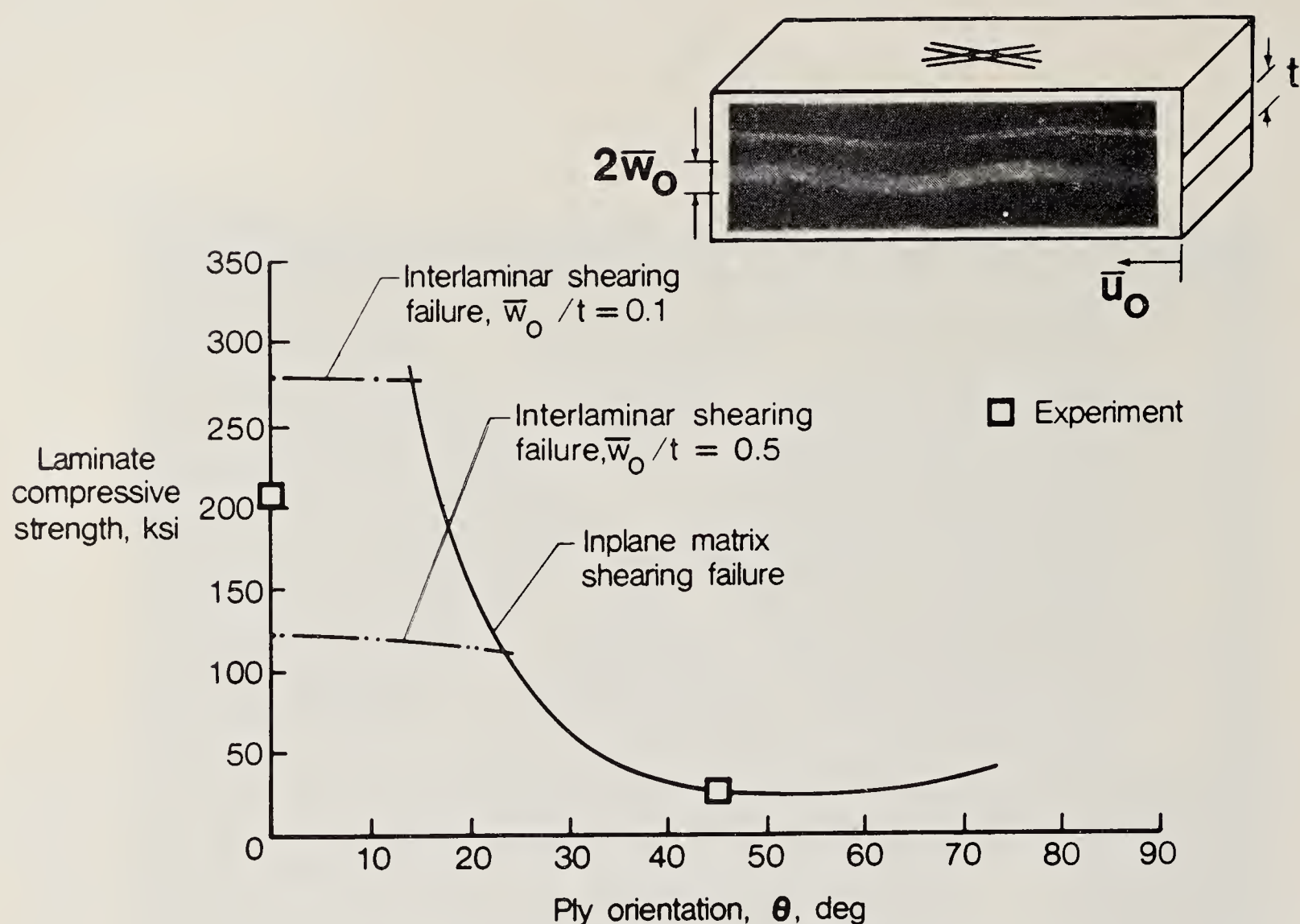


Figure 19. Failure analysis of angle ply laminates.

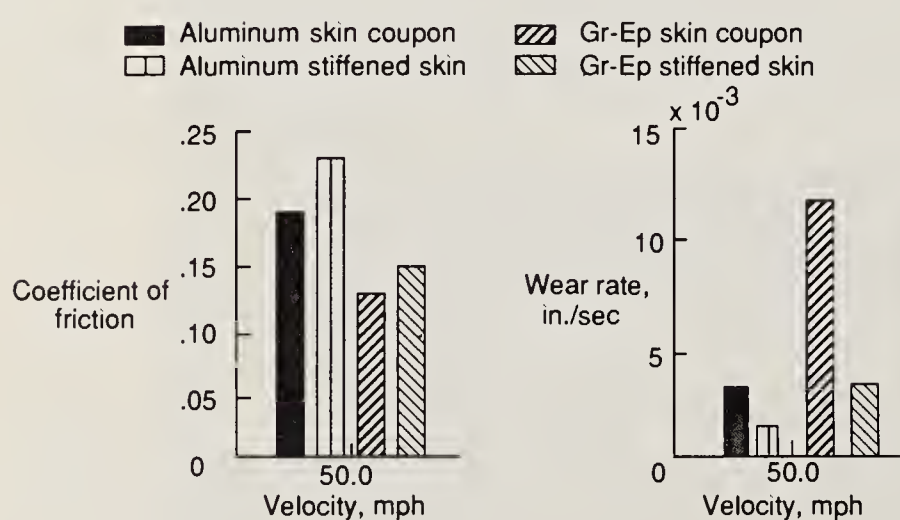


Figure 20. Friction and wear rate data for graphite-epoxy and aluminum specimens.

energy of impact is taken up by plastic deformation of the rings of the fuselage.

A fundamental investigation of the behaviour of graphite fuselage rings is reported in Boitnott & Carden (1986). Results are presented in figure 22. Note that the aluminum ring distorts in a fashion similar to the fuselage shown in figure 21. The composite ring by contrast fails in a brittle fashion. Thus, some designs for energy absorption need to be developed for large composite fuselage structures.

The technology for energy absorption in composite structures is largely being developed in the helicopter industry. An example of recent work is presented in figure 23 (Farley 1987). The figure illustrates that lightweight composite structural elements can be designed to absorb significant amounts of energy.

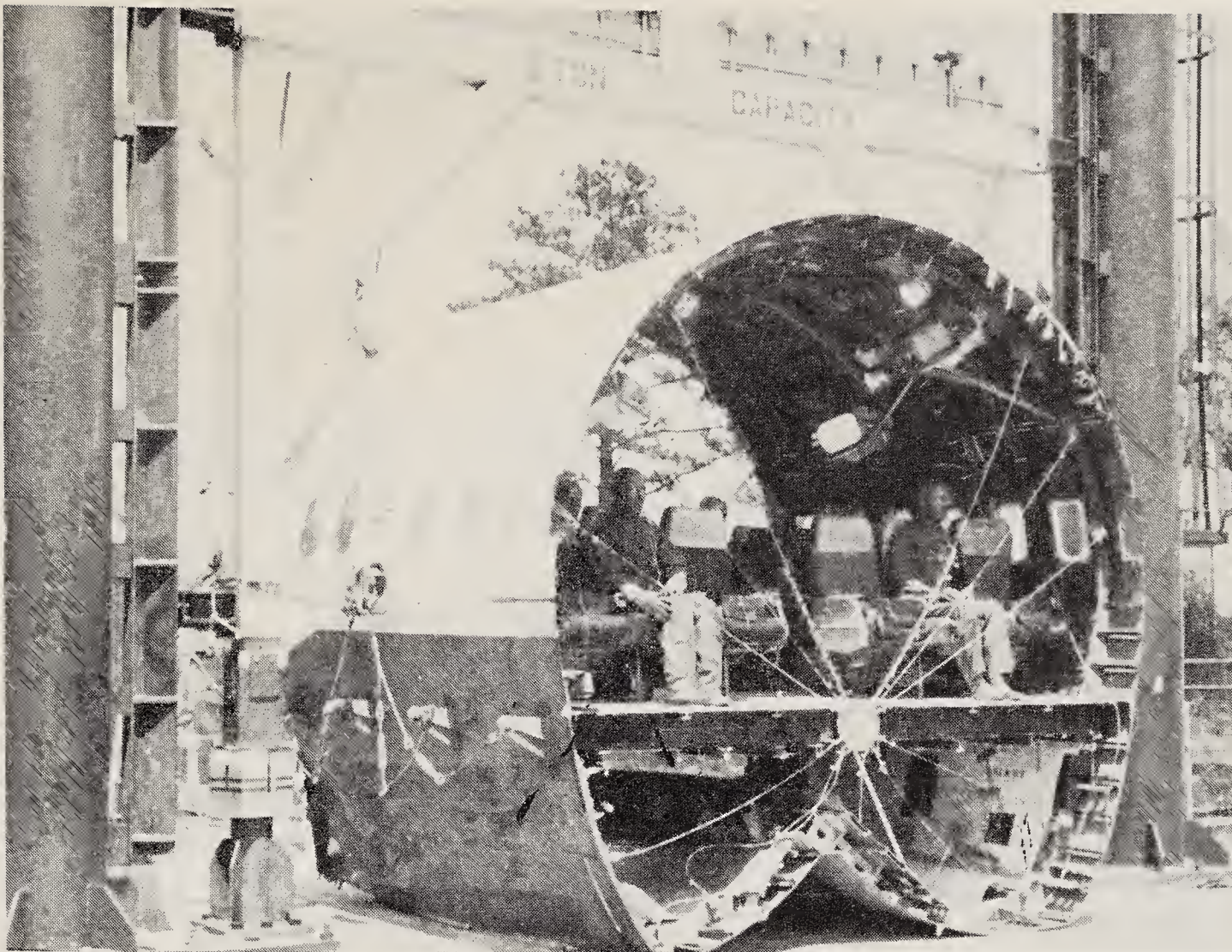
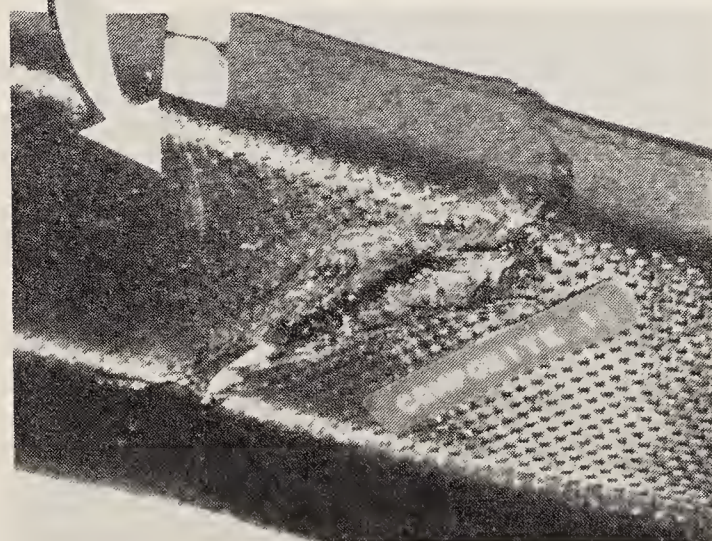
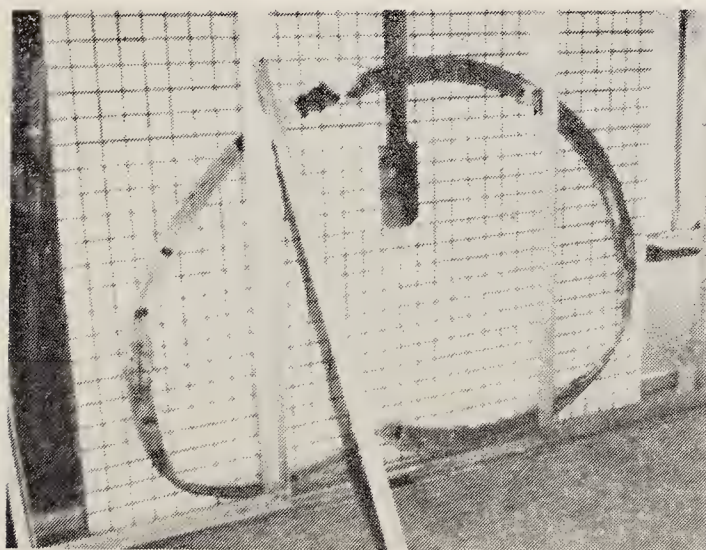
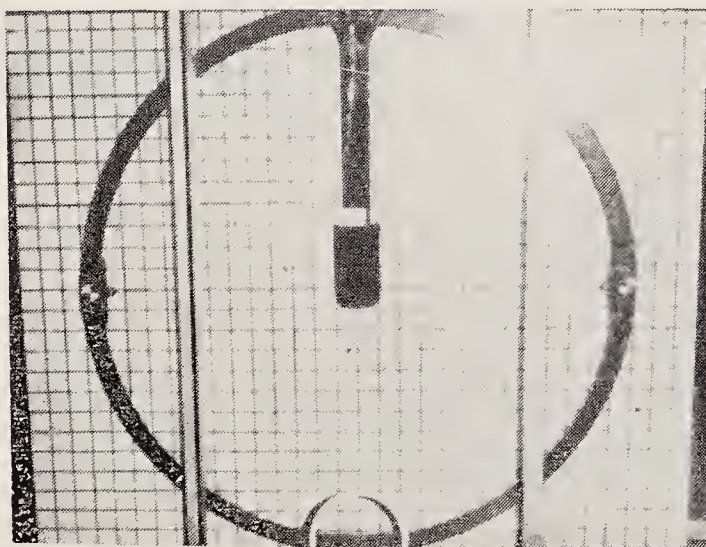


Figure 21. Metallic fuselage shape after drop test.

Composite frame



Aluminum frame

Figure 22. Failure modes of aluminum and composite fuselage ring frames.

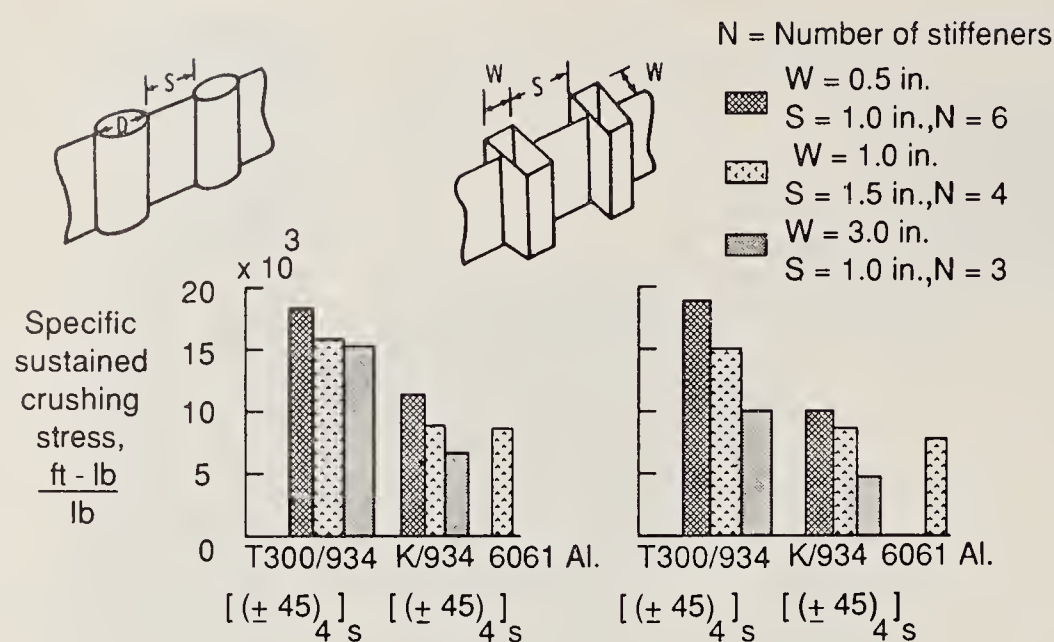


Figure 23. Energy absorption characteristics of composite materials.

8. Concluding remark

Highlights of current research on the mechanics of composite structures at NASA's Langley Research Center have been presented. Lessons learned from NASA's ACEE programme suggest that a better understanding of the local mechanics of complex composite structures is needed. Aircraft designers need better design and analysis tools, verified by experiment, to do their job.

A battery of research analysis programmes exists; however, many of them are general purpose analyses which require sophistication to use and significant computer resources for achieving solutions. The value of special purpose analysis tools has been illustrated by results from an approximate analysis of compressive buckling of a plate with a circular cutout.

A computational structural mechanics programme to address future needs in structural analysis has been described. The programme addresses the problem of global-local analysis in investigations of a complex stiffened panel with an interrupted stiffener. Central to the programme is exploitation of parallel processing machines. An example of two-dimensional vibration analysis on a parallel computer serves to illustrate the potential of parallel computation.

Research in buckling and postbuckling of composites panels has been reviewed. Special purpose tools exist to analyse buckling of stiffened panels under combined loads accurately and efficiently. The ability to predict postbuckling stiffness of composite plates has been developed in general purpose analyses and applied with some success to flat and curved plate experiments. More effort is needed to develop practical approximate formulas for design of stiffened plates with buckled skin.

Progress is being made in identifying new methods of failure in compression-loaded laminates. Transverse shear failures have led to the development of new theories to estimate transverse stresses, stability and postbuckling in multilayered plates. Failure modes observed in angle-ply laminates have generated new studies of short-wavelength buckling. Results from a new failure analysis treating multilayered fibre plates on matrix elastic foundations has shown fair agreement with experiment.

Basic research on the behaviour of composite structures in response to dynamic loading characteristic of fuselage crash loads has been discussed. Wear and friction characteristics have been measured on runway surfaces. Impact loading studies on

composite fuselage rings suggest that energy absorbing concepts will have to be developed to avoid brittle failures of fuselage components.

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Recent research and development, future problems and trends in advanced composite materials in Japan

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Abstract. The progress and contributions of the national research and development project on advanced composites so far are described. Some structural applications of composites in Japan are outlined to examine the structural utility. Future problems and trends are also considered.

Keywords. Composite material; polymer matrix composites; metal matrix composites.

1. Introduction

In order to discuss the recent status of research and development (R & D) of advanced composite materials (ACM) in Japan, it is preferable to first introduce the national R & D project of ACM which is a big, systematic and comprehensive project. The project term (8 years) is from 1981 to 1988 fiscal year. In the present paper, its progress and the contributions so far are briefly outlined, together with current problems. Some rather large size structural applications of composites are then presented to consider structural problems. Current as well as future problems and trend of composites are considered and discussed.

2. National R & D Project on advanced composite materials

In 1981, the Agency of Industrial Science and Technology of the Ministry of International Trade and Industry (MITI), started eight R & D projects for cultivating the seeds of future industries in Japan. One of them was the eight-year R & D project on advanced composite materials including polymer matrix composites (PMC) and metal matrix composites (MMC). Aerospace, automobile and gas turbine industries were taken as the main objective application fields of this project. Since requirements in these leading industries are severe, the results of the project would be expected to give rise to useful ideas for cultivating other industries in future.

Its final development level at the end of the project was planned, by analogy to a “tree”, to take it up to a “young tree” level, starting from the level of “seedling” in 1981.

In the ACM project, a team headed by the present author was organized, consisting of

6 national research institutes, 2 universities and 12 companies, under the leadership and control of the promotion committee and evaluation committee. For concrete promotion and management of the project, the R & D Institute of Metals and Composite Materials for Future Industry was established.

2.1 Objective items for the project

The planned R & D items are as follows:

Target property of advanced composites;

Polymer matrix composites –

- (1) high performance components,
- (2) intermediates: prepregs, 3D-woven preforms,
- (2) fabrication technology;

Metal matrix composites –

- (1) high performance prepregs: wires and sheets,
- (2) fabrication technology;

Evaluation, and its technology;

Design technology.

2.2 Target property of advanced composites

The objectives of the project are to create new high performance ACM and new fabrication, evaluation and design technologies. Among them, in particular for aerospace, it is indispensable to enhance high temperature strength, and these targets, as shown in table 1 and figure 1, were established. It is expected that these target values are attainable but can not be surpassed until the end of the project. PMC and MMC, whose strength values fall within each target area, are desired.

2.3 Polymer matrix composites

2.3a Development of heat-resistant matrix resins (epoxy, polyimide and polyphenyl-quinoxaline) and their composites: (i) Epoxy resins (Report 1983) – three types of epoxy resin systems, highly crosslinked (PGP), hydrophobic (PGN) and heat-resistant (PGI), were studied. Among them, PGP and PGI have shown good mechanical properties at 200° C. In particular, PGI has good long-term thermal stability.

(ii) Polyimide resins: Polyimides (PI) have good thermal stability but have poor fabricability and are expensive. In order to eliminate such demerits and improve the fabricability, while maintaining heat-resistance, three approaches were attempted by (1) selection of terminal group (TPI), (2) modification of main chain (MPI) and (3)

Table 1. Target for advanced composites.

FRP	(1) Heat resistant temperature $\geq 250^{\circ}\text{C}$ (2) Tensile strength* $\geq 2.35 \text{ GPa}$ (240 kgf/mm ²)
FRM	(1) Heat resistant temperature $\geq 450^{\circ}\text{C}$ (2) Tensile strength* $\geq 1.47 \text{ GPa}$ (150 kgf/mm ²)

* Strength by standard specimen and at 90% retention.

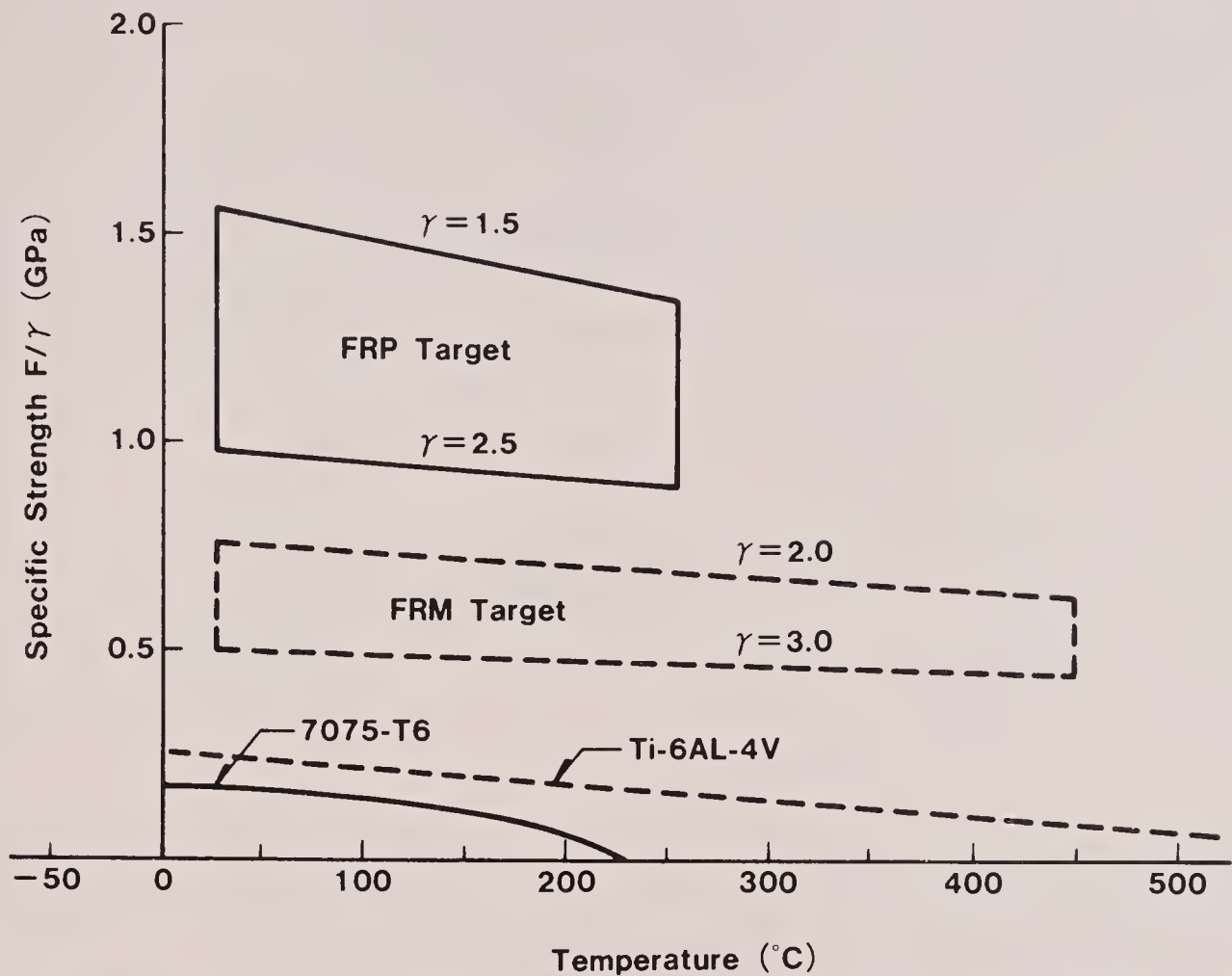


Figure 1. Target area on specific tensile strength versus temperature diagram.

optimization of oligomer structure (OPI).

Table 2 shows some results of such approaches.

In OPI, an attempt to introduce an N-allyl group into PI structure resulting in a novel oligoimide, containing both N-allyl and Nadimide as terminal groups and also containing reactive diluent, which could be cured at low temperature. This OPI-type PI has shown excellent thermal resistance and improved fabricability.

OPI-type PI resin, however, is solid and does not show drapery and tackiness at room

Table 2. Property of polyimide composites*

Item	Temperature	TPI	MPI	OPI	Target for first stage
Tensile strength (GPa)	RT	2.19	1.98	2.33	2.06
	250°C, 10 min	1.94	1.82	2.18	1.86
	-60°C, 10 min	1.84	2.18	2.18	1.67
Tensile modulus (GPa)	RT	164	134	153	152
Elongation (%)	RT	1.34	1.38	1.59	1.35
Flexural strength (GPa)	RT	2.47	2.40	2.38	2.01
	250°C, 10 min	1.82	1.70	1.65	1.53
	250°C, 500 hr	2.43	1.69	1.34	1.32
ILSS (GPa)	RT	0.113	0.067	0.162	0.0882
V_f (%)	—	69.4	64.4	65.0	65

* :Torayca 400/ Polyimide,
 ILSS: Interlaminar shear strength,
 V_f :Fibre volume fraction, (1 GPa = 102 kgf/mm²).

Table 3. Property of torayca 400/PPQ composites.

Item	Temperature	Copolymer PPQ
0° Tensile strength (GP _a)	− 60°C	1.63
	RT	1.63
	250°C	1.71
0° Tensile modulus (GP _a)	RT	151
0° Elongation (%)	RT	1.08
0° Flexural strength (GP _a)	RT	1.47
	250°C, 10 min	1.27
	250°C, 500 hr	1.32
ILSS (GP _a)	RT	0.0882
V _f (%)	—	64.0

temperature. To improve such poor fabricability, the PMR (polymerization of monomer reactant) method, and a small quantity of solvent (e.g. NMP) have been used. It is then possible to make OPI-prepreg cure at low temperaure.

Table 2 shows the excellent properties of T400/OPI composites. However, further modification is needed for OPI resin prepreg for fabrication of complicated structures in autoclaves.

(iii) *Polyphenylquinoxarine* (PPQ) Nadimide-endcapped polyphenylquinoxarine (NPQ) was synthesized. The fabrication of composites was easy due to the high flowability of the NPQ oligomer (see table 3).

Table 4 shows a summary of the properties of improved epoxy and polyimide resins.

2.3b Intermediates, 3D-woven preforms and composites: Mechanical and ablation properties of 3D fabric composites with various constitutions were investigated. Tensile, flexural and shear properties of carbon fibre (CF) 3D fabric/epoxy composites compared very favourably with those of laminates having nearly the same density and fibre volume fraction, except shear strength. Shear strength of 3D-woven fabric composites have shown values greater than four times the values of interlaminar shear strength (ILSS) of similar laminates.

The weaving flexibility of various 3D geometry structural components is being investigated.

2.3c Fabrication technology: It is not too much to stress the importance of fabrication technology in composites. Even if individual components were better, whether good performance as composites could be attained or not would largely depend on the fabrication technology used. In a composite material, it is a peculiar and important character that only when it consolidates by polymerization, its internal structure as a composite material as well as geometrical shape and dimension is completed. Such inherent character should be taken into account in its fabrication technology.

3. Fabrication technology

In the project, the following three ways of R & D were taken up: (1) level-up of

Table 4. Developed resins and their properties.

Resin	Type	Symbol	Specific feature	Thermal decom- position temper- ature (°C)	Laminate tensile strength (kgf/mm ²) test temperature (°C)	Curing temperature (pressure, kgf/cm ²) Aftercure temper- ature (°C)
Advanced epoxy resins	Highly crosslinked	PGP	Glycizylphenolic highly crosslinked epoxy	330	255 (200°C)	180 (6)
	Hydrophobic epoxy	PGN	Naphthalene-hydrophobic epoxy	338	251 (200°C)	190 180 (3.5)
	High heat resistant	PGI	Bis-imide amine curing agent		241 (200°C)	200 180 (20)
						220
Highly heat- resistant polyimide resins	Oligomer PI resin	OPI	PMR-method & N-allyl & Nadimide type PI	417	244 (250°C)	280 (9) 300
	Modified main chain PI	MPI	Modified main chain type PI	451	(250°C)	250 (7) 320
New resins	New resin	PPQ	Nadimide-end capped poly- phenyl quinoxaline	434	210 (250°C)	290 (14)

conventional fabrication technology, (2) development of advanced fabrication methods, and (3) forming technology.

3.1 Level-up of conventional fabrication technology

3.1a Integral fabrication: By autoclave fabrication, various box type and stiffened integral structures were made by lamination under optimum conditions of temperature and pressure which were electrically monitored.

This monitored fabrication technology was developed further and is also being applied to fabrication of heat resistant composites.

3.1b Automatic tape lay-up machine: A numerically controlled automatic lay-up machine, having the following performance, was developed.

Lay-up traverse length: lengthwise—4000 mm
transverse — 500 mm
vertically — 50 mm.

Maximum traverse speed lengthwise: 30 m/min.

Diameter of lay-up roll: 130 mm.

Maximum width of lay-up tape: 76.2 mm.

Laminates made by the machine have shown very good mechanical properties (table 5).

3.1c Low pressure and fast curing fabrication: For the fabrication of large size FRP components for future automobiles, low pressure and fast curing cycle processes are required for high productivity. Figure 2 shows the target for future large size FRP panels.

3.1d Continuous fabrication: (i) Continuous forming of CF/thermoplastics composite profile – Studies on the continuous forming of CF/thermoplastic composite profiles

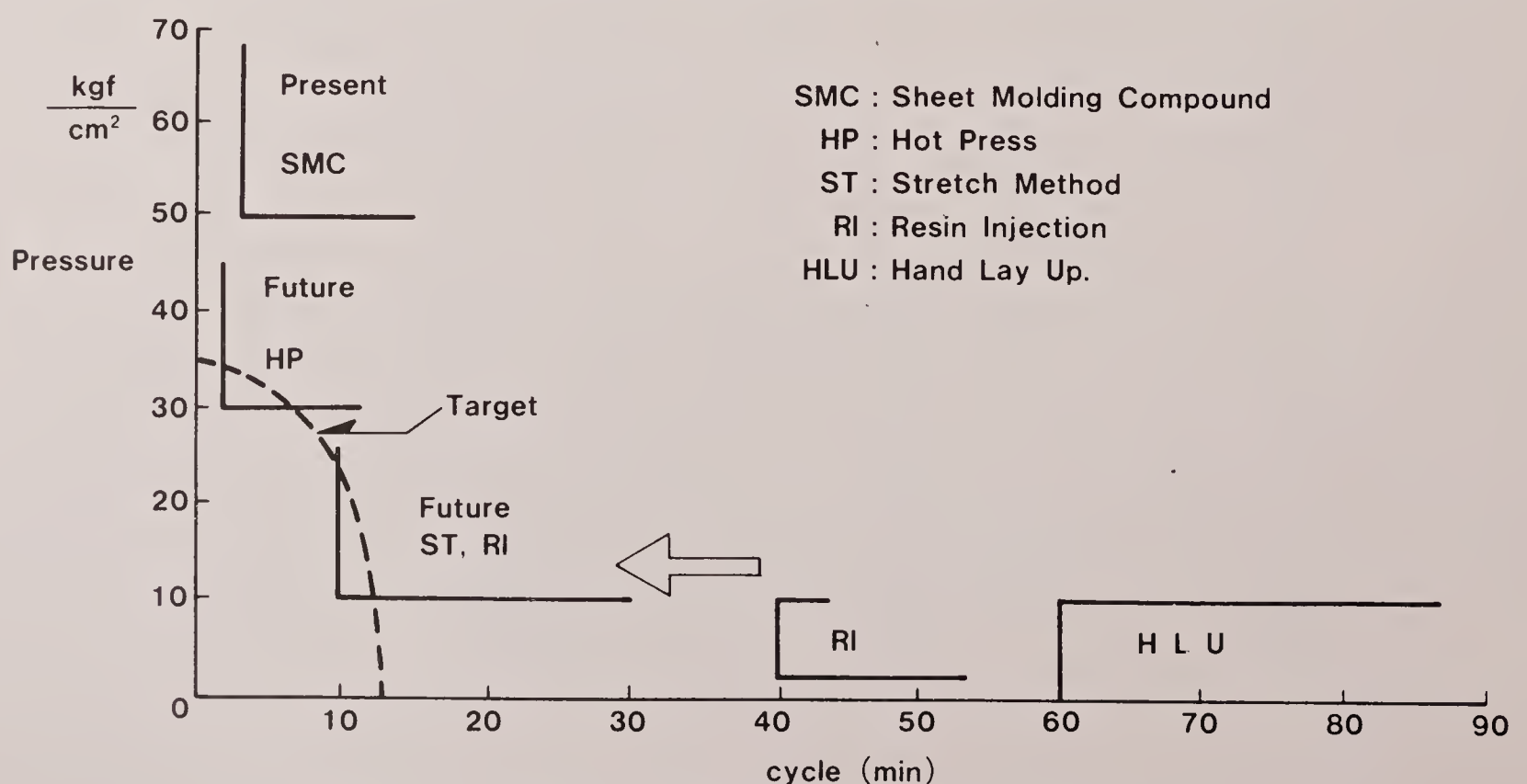


Figure 2. Processing pressure vs. cycle for present and future.

Table 5. Test result of automatic lay-up laminates.

Test piece no	lamination condition			laminates size (B × L) (mm)	Properties test result					
	press force (N)	tape tension (N)	speed (m/s)		ILSS (MPa)		V _f (vol%)		V _v (vol%)	
					L	B	L	B	L	B
1	245	39	0·17	220 × 900	119 (3·9)	115 (1·8)	68·7	68·2	0·2	0·03
2	490	39	0·42	220 × 1,400	116 (2·9)	123 (6·1)	70·8	70·6	0	0
3	490	39	0·42	220 × 3,000	124 (2·7)	119 (3·8)	71·1	71·2	0	0
4	981	39	0·17	220 × 900	127 (2·6)	131 (2·0)	71·7	70·9	0·1	0
5	981	39	0·42	220 × 1,400	122 (1·0)	123 (3·2)	69·4	70·1	0	0
6				220 × 400	127 (1·4)	127 (2·7)	68·6	68·7	0·2	0·2

ILSS = Interlaminar shear strength; Figure inside parenthesis gives C.V., V_f = fibre volume fraction, V_v = void content.
L = fibre direction of UD laminates;
B = transverse direction (90° direction) to fibre.

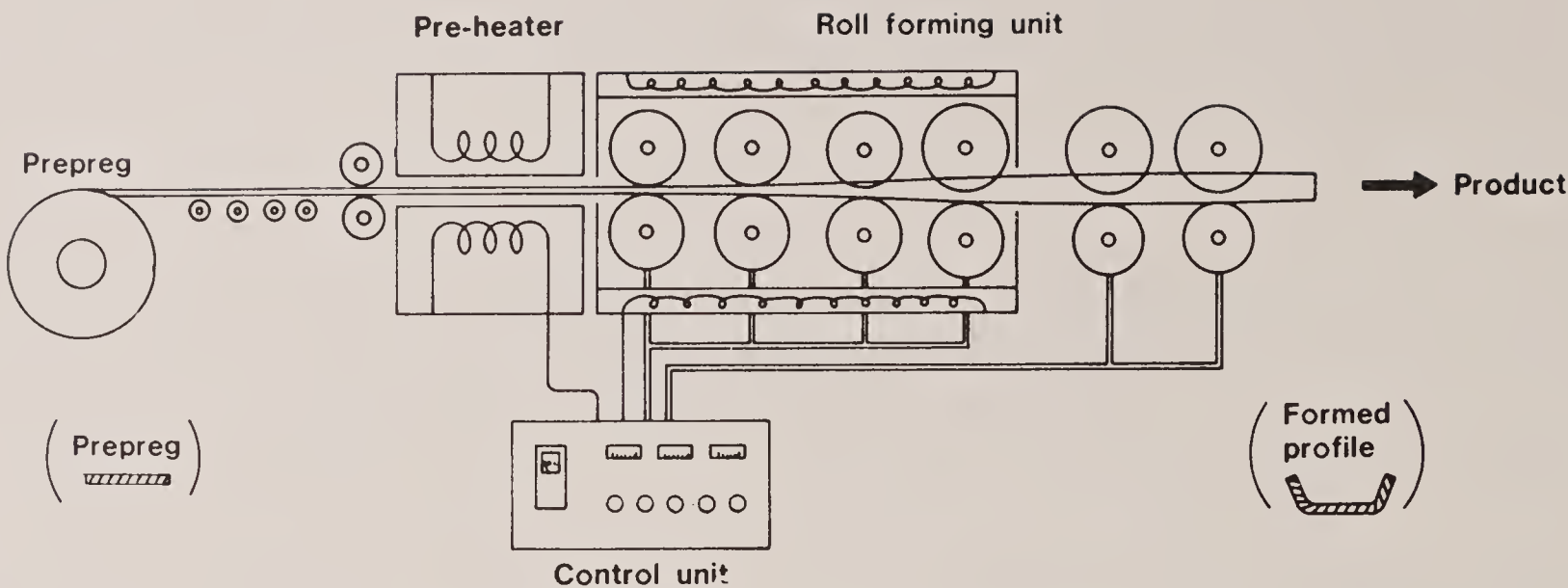


Figure 3. Continuous rolling processing machine.

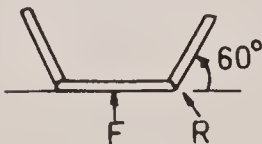
with section roll were carried out for future applications in space structures. Figure 3 shows a schematic view of roll forming machine and table 6 gives the evaluation test results of CF/PES and CF/PEEK made by the facility.

Table 6. Quality test result.
1. Reinforcement and matrix of test specimens

Component		CF/PES	CF/PEEK
Carbon fibre	Type	HM-UD (IM-6)	HM-UD (AS4)
	Number of filaments	12,000	12,000
	density	1.73	1.79
	tensile strength	3920 MPa	3530 MPa
	tensile modulus	284 GPa	228 GPa
Matrix		PES	PEEK

2. Test result

		CF/PES	CF/PEEK
Thickness	mean	0.95 mm	1.12 mm
	c.v.	+ 11.1% - 3.0%	+ 3.6% - 4.5%
V_f at	F	55.2	56.2
	R	56.9	57.2
V_v at	F	13.0	2.8
	R	12.4	2.1
T_g at	F	162	160
	R	158	154
Tensile modulus	GPa	40.2 ~ 49.5	70.7 ~ 77.0
	mean	44.1	74.2
	kgf/mm ²	4100 ~ 5050	7210 ~ 7860
	mean	4500	7570



F and R = measured portion of flat part and bend-forming curved corner, respectively.

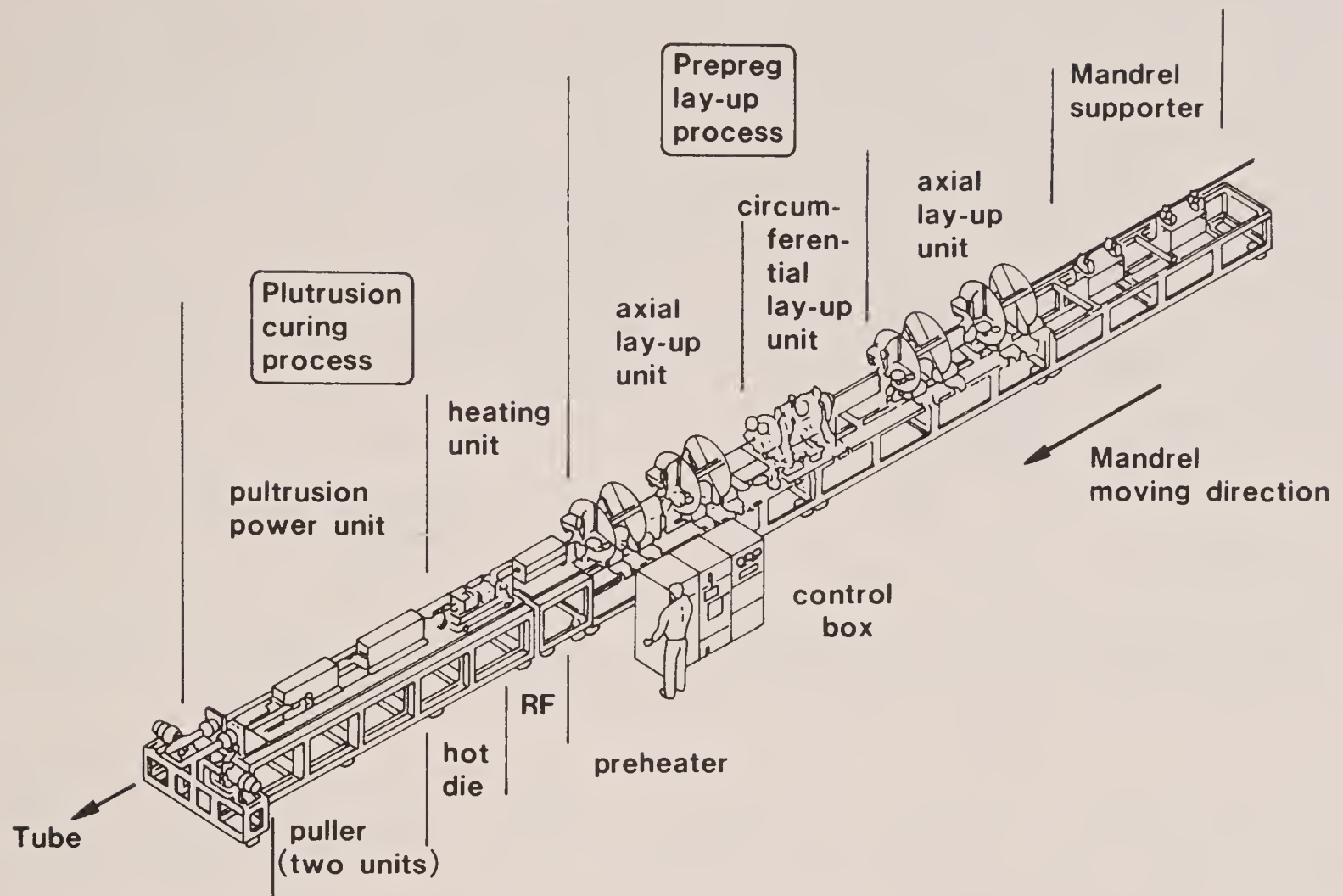


Figure 4. Continuous lay-up pultrusion processing machine for tubes.

(ii) Continuous lay-up and pultrusion forming of thin-walled CFRP pipes – The lay-up machine of CF/epoxy UD prepreg in longitudinal and off-axis directions, combined with the pultrusion equipment provided with a hot-die for continuous curing, was designed and made in order to establish continuous forming process of CF/epoxy thin-walled long pipes. Co-transfer of the mandrel with formed pipes is a peculiar feature of this machine (refer to figures 4 and 5).

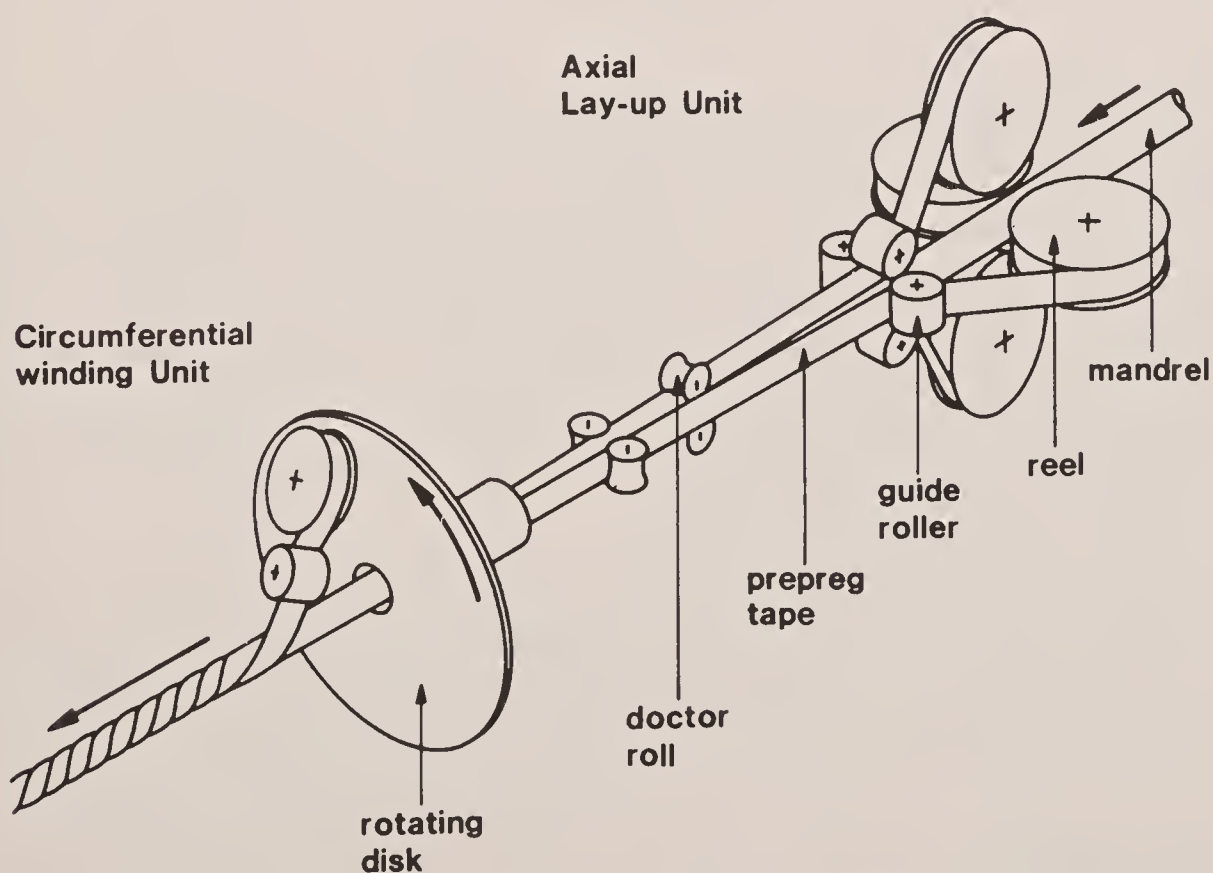


Figure 5. Lay-up mechanism of axial and circumferential preregs.

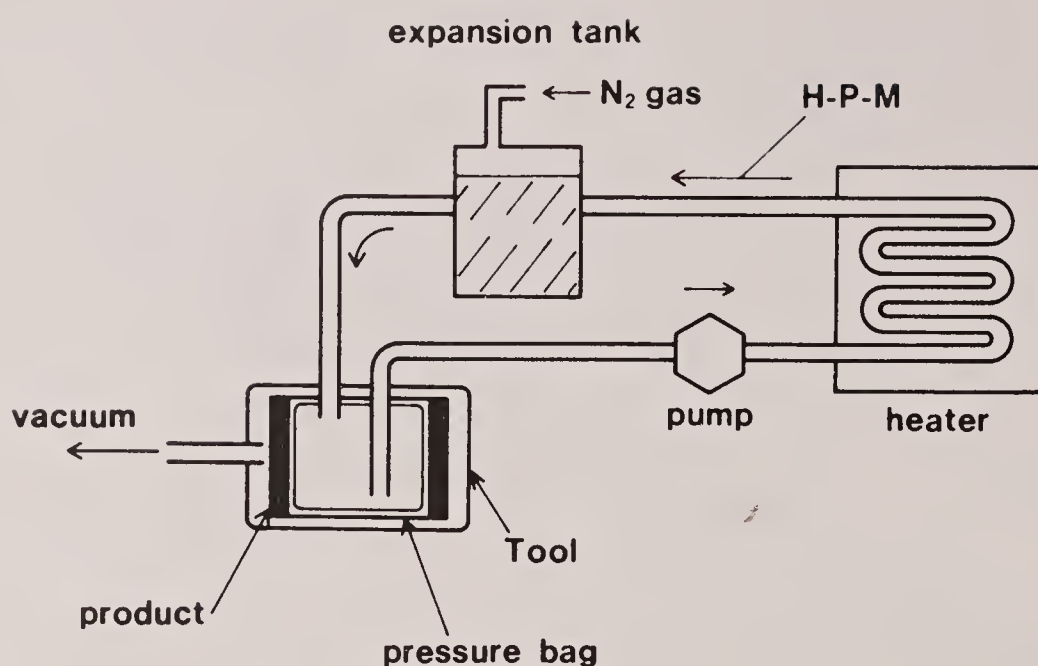


Figure 6. Principle of heat transfer fluid process. HPM: hot-pressure-media.

The thickness of pipes made by this machine varied by about ± 4 to 7%. This has to be reduced and the uniformity of thickness should be improved in future.

3.1e 3D-weaving machine: An automatic three-dimensionally weaving machine was designed. Basic fabrication technology, which involves impregnation techniques for 3D cloth having a tailored shape, and a curing process to realize high performance as composites, was developed.

3.2 Innovative development of new fabrication technology

3.2a Heat transfer fluid process: Autoclaves have been widely used for curing of composites. This has several advantages, but limits the size of products and consumes a huge amount of heat energy. In order to eliminate such demerits, a new manufacturing technology, which does not limit the size and has high efficiency of heat transfer, has been developed. The feature of this method is to use fluids of high temperature and high pressure suitable for curing of composites. CF/epoxy cylindrical test specimens have been successfully fabricated by this method. Some results are shown in figure 6 and table 7.

3.2b Microwave curing process: In order to obtain highly efficient curing of composites, a microwave curing process was investigated. Several thermoset resins and their prepreps were cured by microwave heating equipment which consists of a magnetron source at 2.45 GHz and a power supply control device, and the curing characteristics of the products have been studied. It was found that power consumption is low, uniform heating and then uniform polymerization are obtained, and curing time is sufficiently reduced, compared with the conventional method. The tooling and equipments were also developed.

3.2c Electro-magnetic planting process of short fibres: A fabrication method for increasing interlaminar shear strength of laminates was developed, in which metal-plated chopped fibres are planted electro-magnetically in between laminae perpendicular to the laminate surface.

3.3 Forming technology

Integral configuration design, avoiding bits-assembly design of products, should be preferred as far as possible. But a shape-up forming process is always necessary, for

Table 7. Measurement of dimensions of stiffened cylinders fabricated by autoclave method and heat transfer fluids process, showing close accuracy with each other.

Measured dimension		Design dimension	Auto-clave			Heat transfer		
			A	B	C	A	B	C
			333.8	332.8	332.9	333.1	333.3	333.2
Outer diameter		333.2						
d		(mm)	332.8	+ 0.7 − 0.3		333.2	+ 0.3 − 0.4	
			21.3	21.6	21.3	21.3	21.9	21.4
Height of hat		21.6						
h		(mm)	21.4	+ 0.4 − 0.4		21.5	+ 0.5 − 0.3	
			1.55	1.73	1.54	1.53	1.68	1.52
Thickness	t_s	1.6						
(mm)	(1)		1.61		+ 21.7% − 8.7%	1.57		+ 14.0% − 10.8%
			1.96	2.05	1.94	1.95	2.12	1.96
	t_{st}	2.1						
	(2)		1.99		+ 9.0% − 4.5%	2.01		+ 14.4% − 7.0%
			1.94	2.04	1.87	1.94	2.37	2.00
	t_{sh}	2.1						
	(3)		1.95		+ 12.8% − 10.3%	2.10		+ 16.7% − 9.5%
			0.76	0.75	0.74	0.74	0.80	0.77
	t_{hw}	0.9						
	(4)		0.75		+ 6.7% − 6.7%	0.77		+ 10.4% − 9.1%
			0.74	0.75	0.72	0.73	0.74	0.75
	t_{hc}	0.9						
	(5)		0.74		+ 8.1% − 5.3%	0.74		+ 8.1% − 5.4%

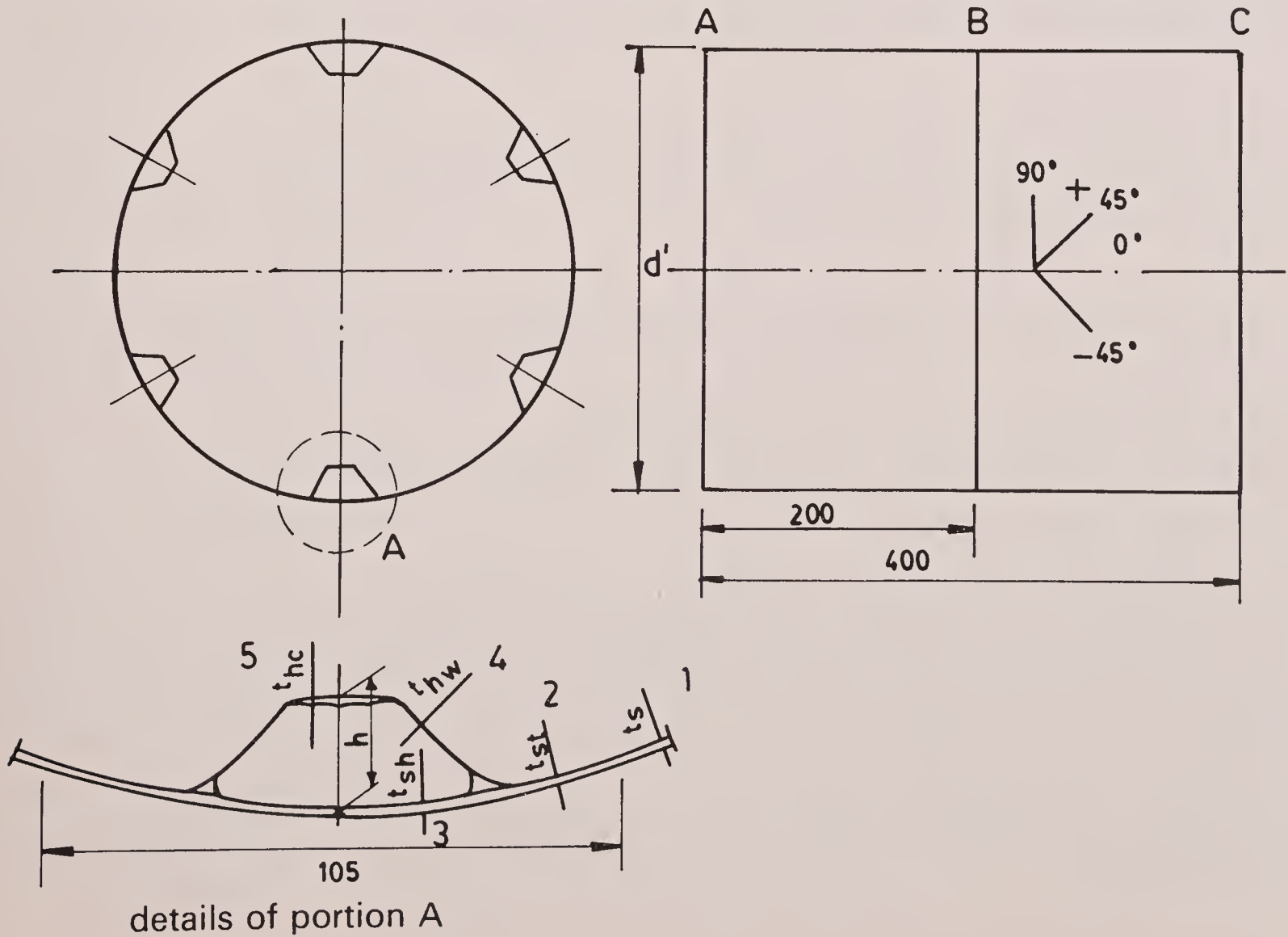


Table 8. Development of fabrication technology for PMC

1. High level-up of performance, reliability and productivity of conventional fabrication technology.

Method	Application Field	Results obtained
a. Integral lamination	Aircraft	Development of electrical monitoring technology of curing of integral lamination; Study of its extension to heat resistant matrix case.
b. Automatic lay-up	Aircraft	Development of basic technology of automatic lay-up machine head design for plane and cylindrically curved plates.
c. Low pressure fabrication	Automobile	Fast curing resin injection fabrication technology, CF/GF hybrid floor panel of cars. Press stretch forming technology for body panel
d. Continuous fabrication	Space	Hut profile continuous-bending fabrication facility due to multi-roll for CF/PEEK. Continuous drawing fabrication facility for CF/epoxy thin-walled tubes.
e. 3D-weaving	Space	3-dimensional automatic weaving facility

2. Development of new fabrication technology

f. High temperature fluid pressure fabrication	New fabrication method in which fluid is used as circulating heat and pressure media (anti-autoclave method).
g. Micro-wave curing process	Development of fabrication method using internal heating by micro-wave.
h. 3D Woven cloth/epoxy composites	Development of basic fabrication method which involves 3D cloth tailored shape impregnation and curing process so as to give high performance.
i. Electro-magnetic Planting and Pressing Process	Development of fabrication method for increasing interlaminar shear strength of laminates in which metal-plated chopped fibres are vertically planted electro magnetically in between lamina.

References: Preprints of the Symposium on Composite Materials for AIST R & D Project for Future Industry in 1933–1986, sponsored by Research and Development Institute of Metals and Composites for Future Industry and Sangyo-gijutsu Shinko-kyokai.

example, in finishing, machining, and surface treatment etc. These technologies were investigated in the earlier stage of the project. The processes of lathe cutting, water-jet cutting, ion-plating and so on were investigated.

Table 8 gives a summary of the work on fabrication methods.

3.4 Metal matrix composites

In the project, the emphasis was mainly placed on the R & D of metal matrix composites (MMC) of aluminium and titanium matrices, using local carbon fibre (CF), and polycarbosilane silicon carbide fibre (SiC). Uni-directionally solidified alloys were not included in our project.

For MMC, our objectives were R & D of high performance preforms, fabrication technology and evaluation technology as mentioned before. The main result is presented here.

3.4a Interfacial problems: In the fabrication of MMC, high temperatures and heating rates are always needed, and in order to enhance performance, careful consideration is required of the problems of interfacial diffusion, control and suppression of heat, and good compatibility between fibre and matrix.

Compatibility problems between CF and Al, SiCF and Al, CVD-fibre and Ti, and so on were investigated.

In our project, the studies have been promoted on the above compositions. For example, in the case of monofilament composites made by the PVD technique, it was found the addition of V or Al in the Ti matrix is effective in reducing the reaction in the interface. In Ti reinforced by rather thick CVD-fibres of diameter about 142 micron such as B/W, B₄C.B/W, SiC.B/W, SiC/C etc., the interfacial reactions were experimentally checked.

3.4b *R & D of high performance wire preforms*

In the case of the Al matrix, a bundle of CF or SiCF filaments, which were infiltrated with melted aluminium, is called "preform wire" or "wire preform" of CF/Al or SiC/Al. As a wire preform is a bundle of multi-filaments coated with Al, it behaves as a bar of UD-composite material. In the fabrications of MMC which is made from wire preform, it is first necessary to make wires of good quality.

(i) *CF/Al and SiC/Al wire preforms* Using two kinds of CF: high modulus CF(HM-CF)-Toray M40 and high strength CF(HT-CF)-Toray T300 and Nicalon SiC fibre as reinforcement, and four kinds of aluminium: 1080 (pure Al), 6061 (1% Mg, 0.5% Si), 5052 (2.5% Mg) and AC4C (7% Si) as matrices, wire preforms were fabricated by the liquid infiltration process. High temperature tensile tests for these wire preforms were carried out. In such tests, however, the fracture can occur very easily at the chucked portion of a specimen. In order to avoid such fracture, a new test method was attempted, in which using wire specimens of 280 mm length and a heater of 100 mm length, only the central portion of about 100 mm length of the specimen was heated, under the following test conditions:

heated portion = central part of 100 mm length,
 heating time = 10 to 15 min,
 chuck distance = 200 mm,
 test speed = 2 mm/min,
 chucking device = air chuck of 65 mm length, using soft asbestos sheet and emery paper.

The tests were successfully conducted with the results as shown in figures 7–9. Figures 7a–d show the high temperature tensile strengths of high modulus M40 CF reinforced aluminium and figure 7e shows tensile strength vs. soaking time at 450° C. From figures 7a–c and e, it is seen the tensile strengths at 450° C have reached about 1.3 to 1.4 GPa. Figures 8a–c represent somewhat similar strengths for Nicalon SiC/Al. Figures 9a and b give the case for HT-CF T300/Al. As the HT-CF has high reactivity, they were coated with carbon by chemical vapour deposition (CVD) method.

Thus CF/Al wire preforms having good high temperature strengths close to target values were obtained.

(ii) *CVD-SiC fibre reinforced Ti alloy* – Applying the CVD method on CF as core, so-called CVD. SiCF fibre is obtained. Here it is designated simply as "SiCF". Such SiCF fibres

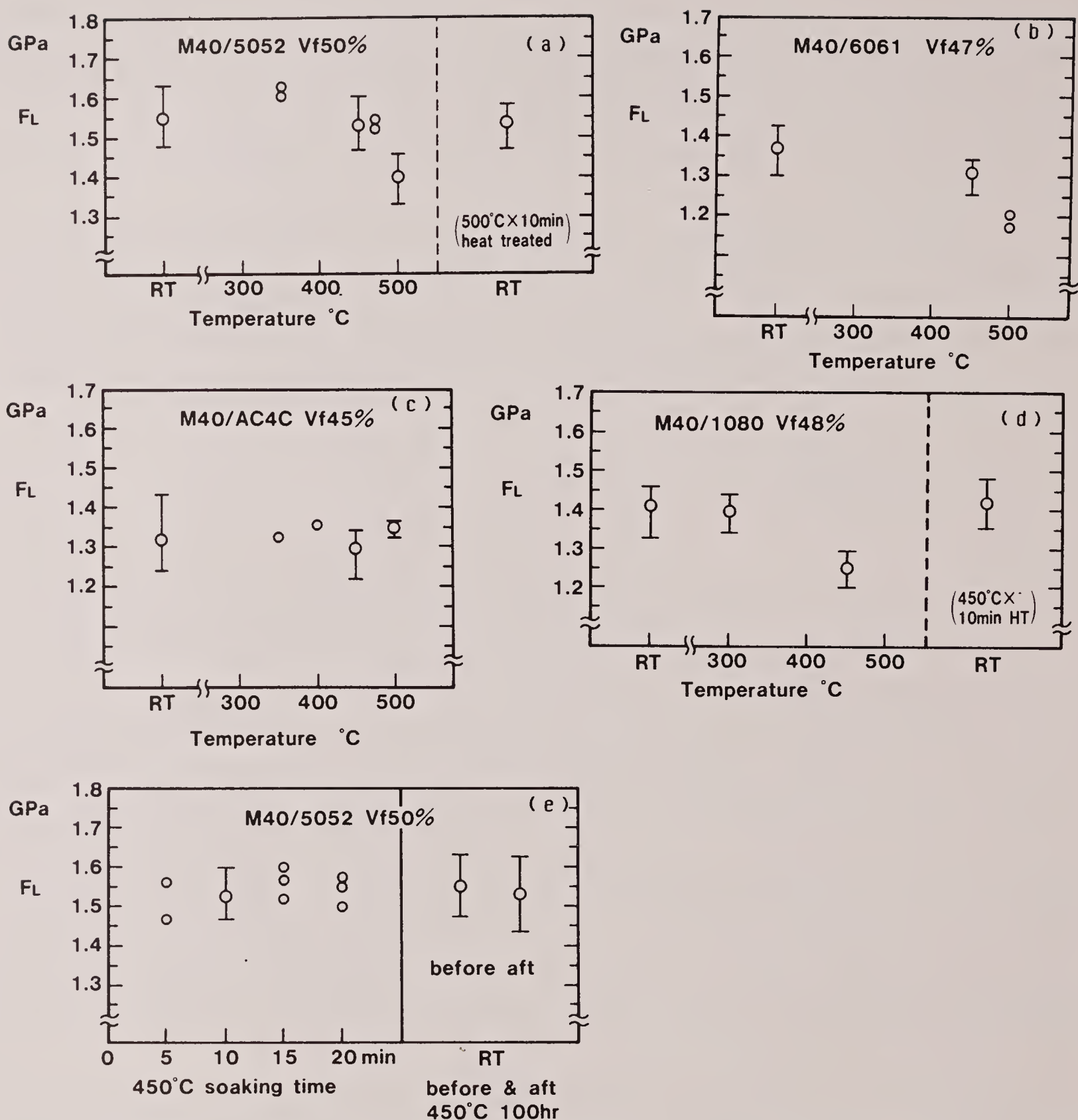


Figure 7. (a) High temperature strength of HMCf. M40/5052-Al. F_L = tensile strength (GPa). RT = room temperature. Vf = fibre volume fraction (%). (b) High temperature strength of M40/6061 aluminium. (c) High temperature strength of M40/AC4C aluminum. (d) High temperature strength of M40/1080 aluminum. (RT = room temperature.) (e) High temperature strength of M40/5052 Al vs. 450°C soaking time.

have high stability for Al and Ti and hence their wide application is expected. SiCf/6061 Al, SiCf/AlFeCe and SiCf/Ti-6Al-4V were made by the hot-press process, and their high temperature tensile strengths were tested. Table 9 shows the retention % of tensile strengths at 300°C and 450°C, compared to room temperature strength values. It was found that these alloys are of considerable high strength, which is dependent on matrix property (table 9).

3.4c Fabrication technology: (i) *Fabrication of SiC whisker/Al composites* – In the fabrication of rods of SiC-whisker reinforced Al, an economical process was developed. This method consists of three processes: (1) fabrication of whisker preform, (2) fabrication of billet of whisker and Al by press-impregnation of melted Al into whisker preform and extrusion of the billet to a rod or final product through moulds. For

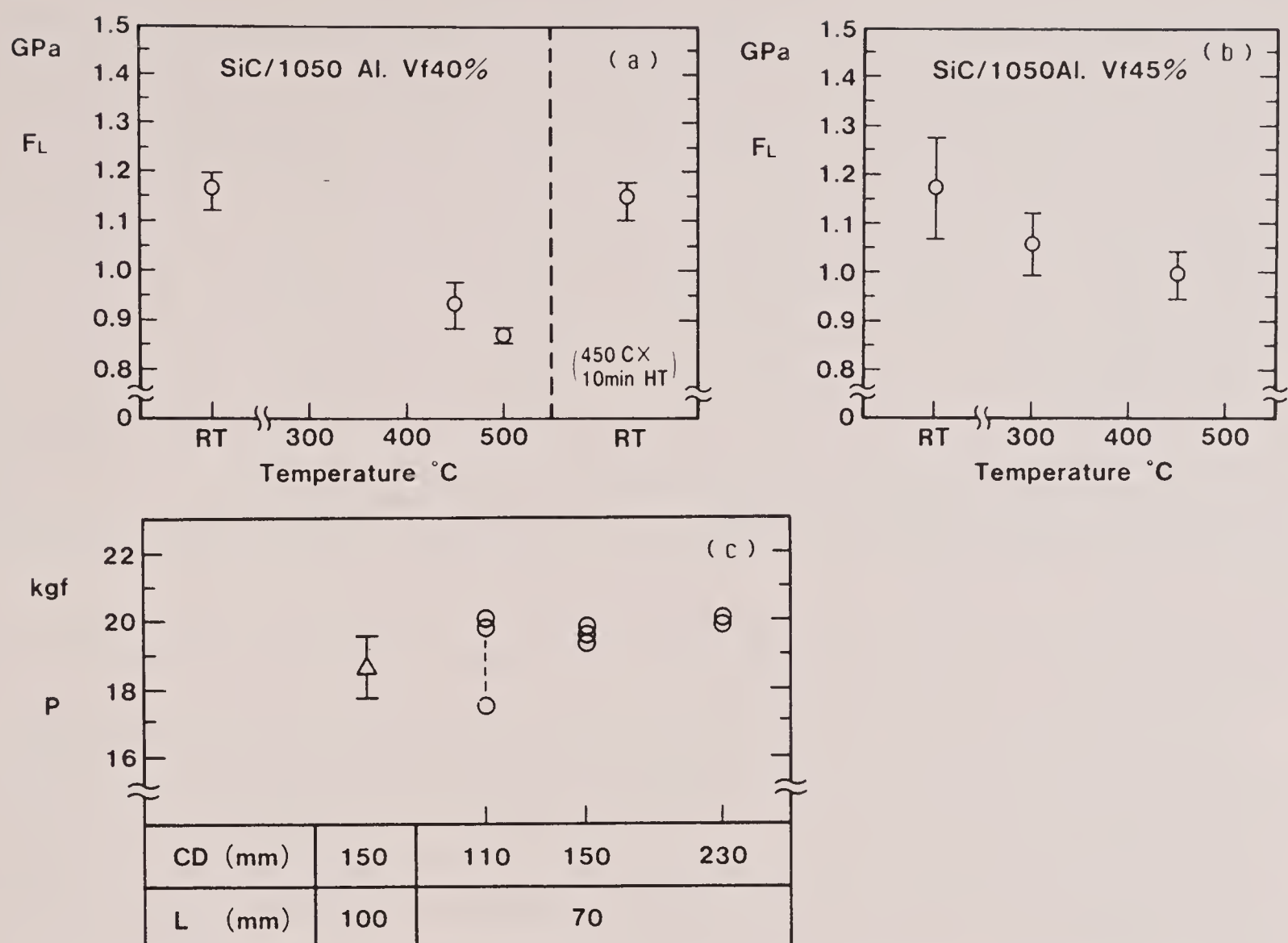


Figure 8. (a) High temperature strength of Nicalon. SiC/1050 aluminum (b) High temperature strength of Nicalon. SiC/1050 aluminum. (c) Tensile strength (P) of Nicalon. SiC/1050 Al at 450°C for various chuck distances (CD) & furnace lengths (L). (N_2 gas used in 100 mm length furnace, No N_2 gas used for 70 mm furnace.)

making whisker preform, SiC whisker is uniformly dispersed into warm water and its slurry is poured into a hollow metal tube, by compressing which whisker preform is obtained. SiC whisker/Al composites made by this process have smaller specific strength and stiffness, but these values are expected to be increased in future.

(ii) *Fabrication of CF/Al composites by hot isostatic pressing (HIP)*—In MMC, as its secondary machining is usually hard, near net shape fabrication is desirable. For this a powder metallurgical method, namely HIP process, for composites was studied. Up to

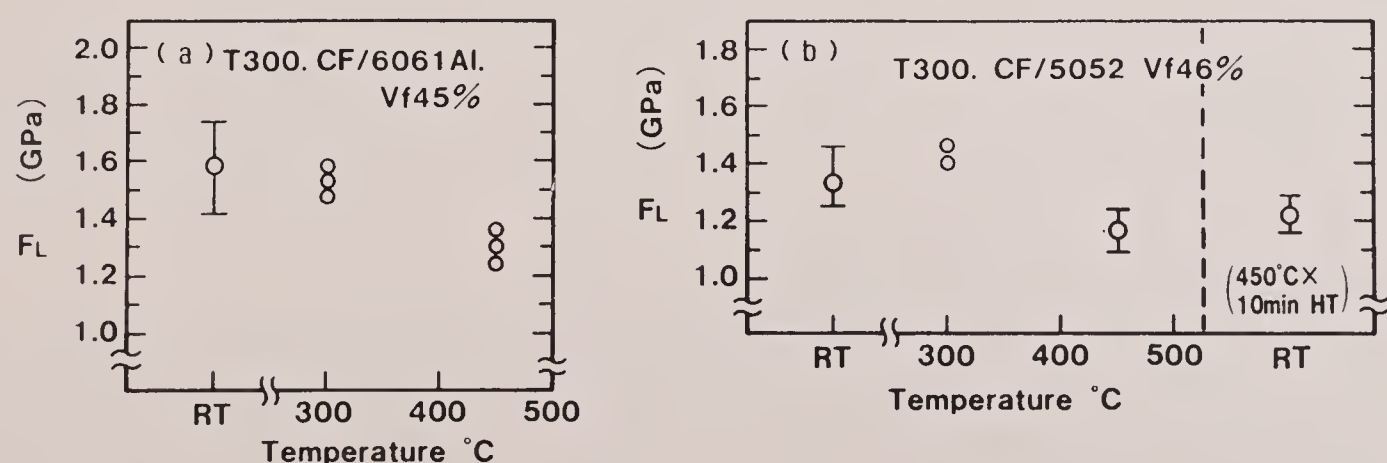


Figure 9. (a) High temperature strength (F_L) of CVD. C-T300 CF/6061 Al. No N_2 gas, 55 mm length electric furnace, chuck distance = 150 mm, soaking time = 10 min. (b) High temperature strength (F_L) of CVD. C-T300 CF/5052 Al.

Table 9. Retention of tensile strength of various CVD. Fibre SiCF/metal matrix composites at 300 and 450° C.

Specimen	$V_f\%$	F_L GPa	Retention %*	
			300° C	450° C
SiCF/6061 Al	50	1.56	90	74
SiCF/6061 Al	37	1.33	86	75
SiCF/AlFeCe	40	1.55	100	102
SiCF/CF/Al**	40 + 12 CF	1.58	90	76
SiCF/Ti (6Al, 4V)	43	2.03	93	93
SiCF/Ti (6Al, 4V)	34	1.82	90	90

* Retention = ratio of high temperature strength divided by room temperature strength,

** SiCF/CF/Al = hybrid composites of SiCF, CF and Al. V_f of these components: 40, 12 and 48%, respectively.

date, whisker or short fibre reinforced MMC have been investigated, but HIP studies in continuous fibre composites are few.

In this study, as a matrix having a low melting point, 4032 Al powder was selected, and argon gas-atomized Al powder was used for protection of its oxidation. In order to uniformly disperse the powder into the CF bundles, the slurry bathing process was used to prepare the preform. Cutting the slurry preform into required lengths and using graphite forming moulds, CF/4032 Al powder composites were made by the vacuum hot press process.

The tensile strength of UD-HIP composites-CF/4032Al, dependent on powder atomized size and its volume fraction, was about 840 to 910 MPa which is still too low.

Hence, further work on it is necessary.

(iii) *HIP processing of CVD fibre reinforced titanium composites* – The fabrication by HIP of the fibre reinforced titanium alloys with B, B₄C, SiC filaments produced by CVD was investigated. Because of limitations in available foil-shaped titanium alloys, powder pre-alloyed by the rotating electrode process was used to change the matrix alloy.

The optimum fabrication condition and combination of fibre and matrix alloy were selected by studying micro-structure, mechanical properties and fractography.

Tables 10a and b show the reinforcement and matrix materials of test specimens. Figures 10 and 11 show the effect of fabrication temperature on tensile strengths of SiC (CVD) reinforced titanium alloys made by the HIP process. The tensile strengths also depend on fibre and matrix materials, as shown in figures 12 and 13.

It can be seen from the above results that high temperature strengths of SiC (CVD) reinforced titanium alloys made by the HIP process are higher than the target values. Table 11 gives a brief summary of R & D of metal matrix composites.

3.5 Evaluation and evaluation technology

The R & D work in the project have been carried out by participating companies and institutes. The results have been periodically evaluated every three or two years by the evaluation committee, whose suggestions have been incorporated in the successive stages of R & D. The cross check for material testing was also performed between the two facilities. (Report 1983; Hayashi 1985.)

The evaluation for the level of R & D achieved as compared with the target was

Table 11. Research and development of metal matrix composites.

a. Development of preforms.

Fibres	Surface treatment	Symbol	Matrix	Preform
Whiskers	coat infiltration	SiC SiC _w	Al	“billet” for extrusion
CF(pan, pitch) SiC (pcs)	coat infiltration	CF SiC	Al Ti	CF wire preform of 100 m length F _L = 1·20 GPa.
CVD-Fibres	coat infiltration plasma spray	SiCF	Al Ti	SiCF/Al or Ti F _L = 1·86 GPa at 450° C

b. Development of fabrication methods.

Method	Specific feature
Liquid infiltration	Most conventional processing CF/Al, SiC/Al, SiC/Ti, CVD-Fibre/Ti.
Press, roll extrusion	Whiskers, short fibre MMC. slurry, extrusion etc.
MOCVD	Low temperature processing, etc.
HIP	(1) HIP: slurry method HM-CF/Al, HT-CF/Al etc. (2) HIP: under high gas pressure and high temperature for CVD-Fibres, F _L = 1·4 to 1·6 GPa SiCF/Ti-6Al-4V, F _L = 1·7 GPa SiCF/Ti-6Al-2Sn-4Zr-6Mo at 450°C.

- carried out based on the following:
- (i) Resins, prepregs and 3D woven clothes,
 - (ii) Fabrication technology:
fabricability, improvement of curing condition by monitoring, void content, fibre alignment, interlaminar shear strength, new fabrication methods, fast curing process, etc.
 - (iii) Test methods:
test methods at high temperature and space environmental conditions, x-ray, AE and NDT methods,
 - (iv) Mechanical properties of composites:
modulus, tensile strength, ILSS, elongation, fracture toughness, etc.
 - (v) Evaluation of quality and reliability of newly developed composites.

With the limited time and budget, the scale of R & D was inevitably unsatisfactory. More comprehensive evaluation tests of the composites developed are necessary.

3.6 Design problems

In the design of composite structures, apart from “bits-assembly designs”, integral

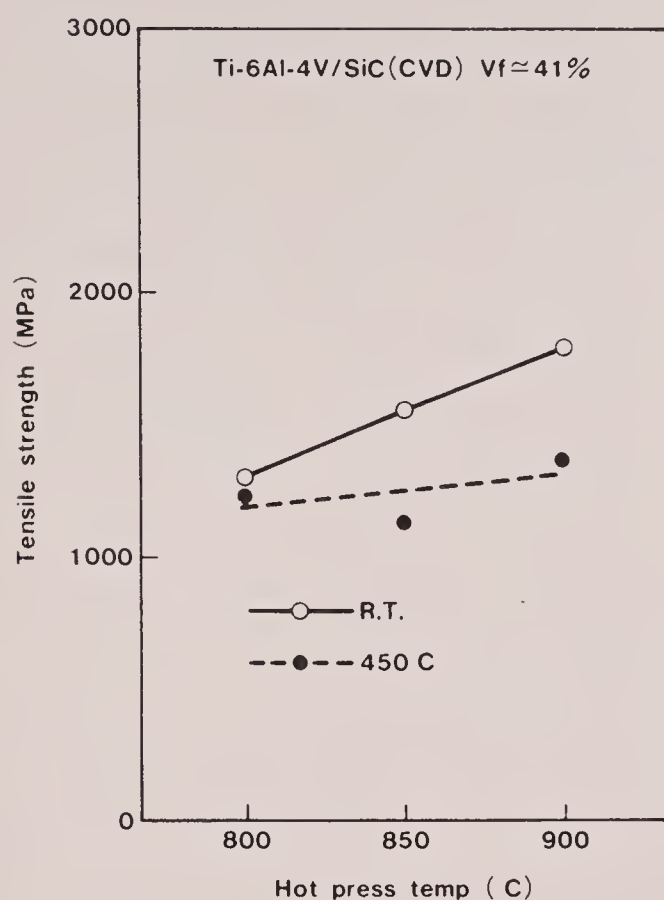


Figure 10. Effect of prefabrication temperature on tensile strength of SiC (CVD) Ti-6Al-4V.

structure – jointless or with fewer joints should be strongly recommended. (Report 1983; Hayashi 1985.)

Optimum design parameters for strength and vibration of blade-like structures, box type wings and space trusses were analyzed by FEM and tested. The analyses and tests have shown good agreements for strength and eigen-frequency.

From study of compression panels, it was found that the truss core type sandwich panel is of the highest strength by weight.

The fabricability of an integral structure of a box type multi-cell wing was examined. In spite of its very complicated structure, low void content and good quality were obtained using well-designed moulds and flexible core materials.

3.7 Level of achievement of the project target

As a whole, the level of achievement of the project as of March 1987 is 85% or more.

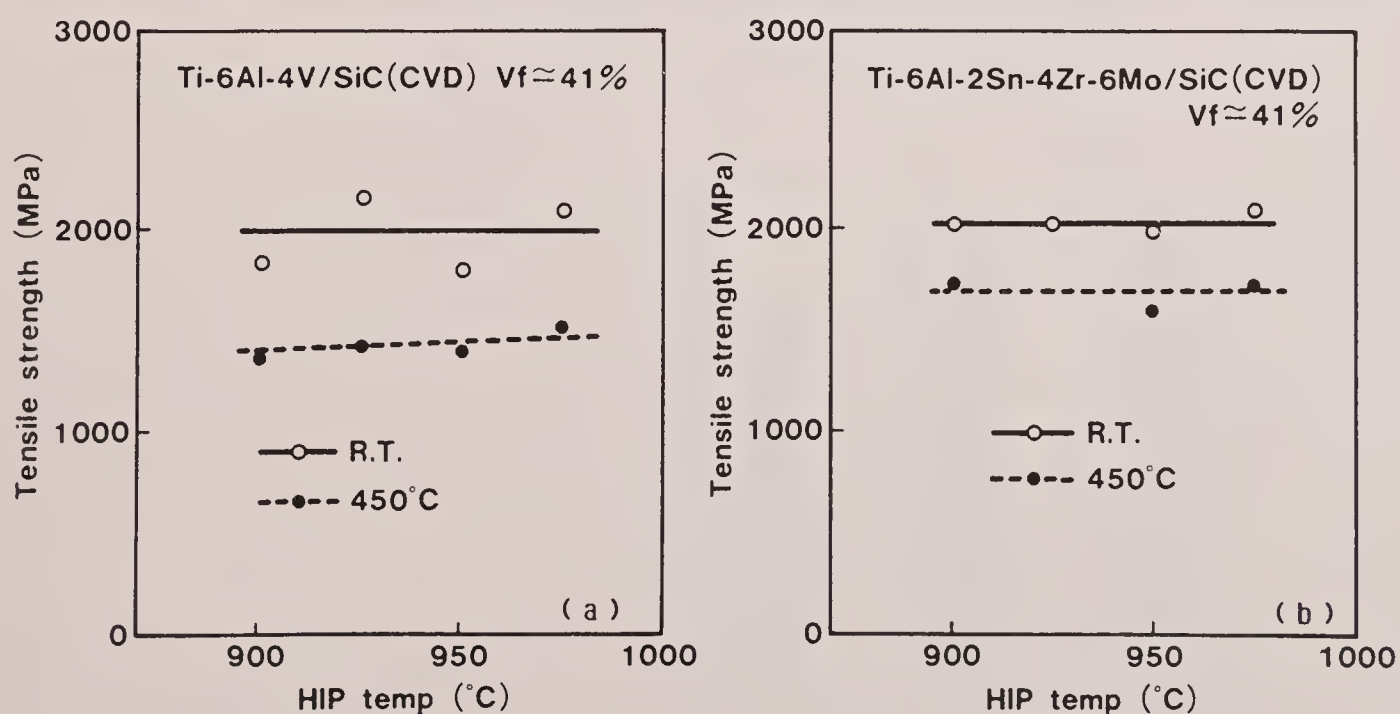


Figure 11. Effect of fabrication temperature on tensile strength of (a) SiC (CVD)/Ti-6Al-4V and (b) SiC (CVD)/Ti6Al-2Sn-4Zr-6N.

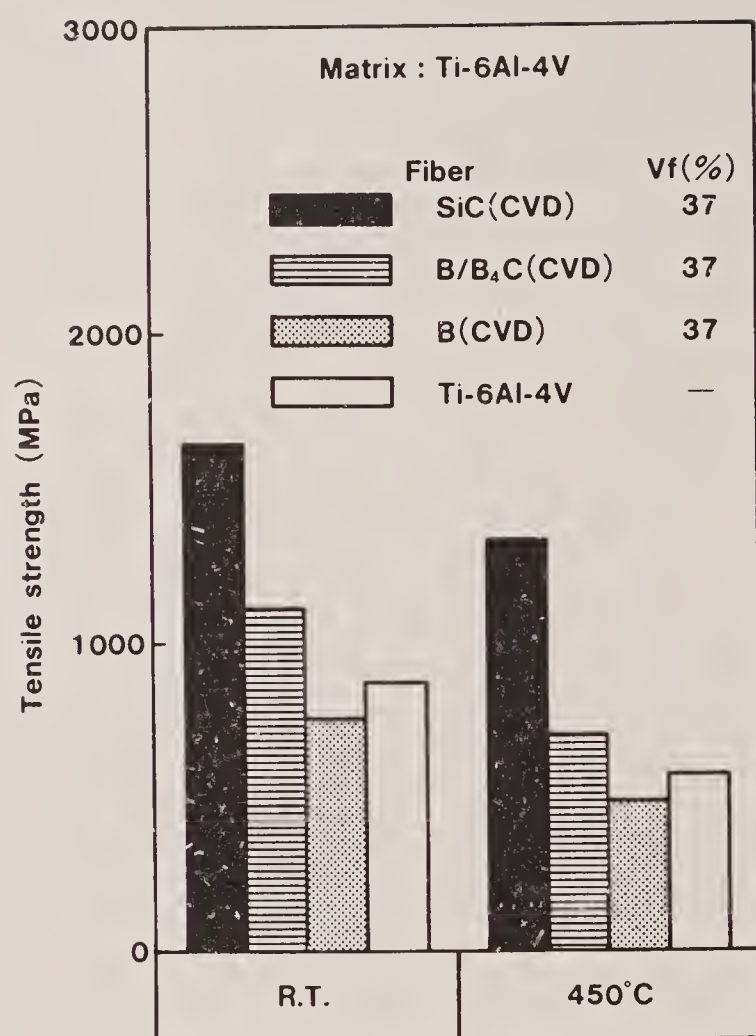


Figure 12. Effect of fibre on tensile strength of composites.

For example, test data of newly developed composites having high temperature resistant resins as the matrix are shown in figure 14. It shows that test data are close to target values.

In addition, various new innovative technologies, as mentioned above, have also been sufficiently developed over the whole area of the project.

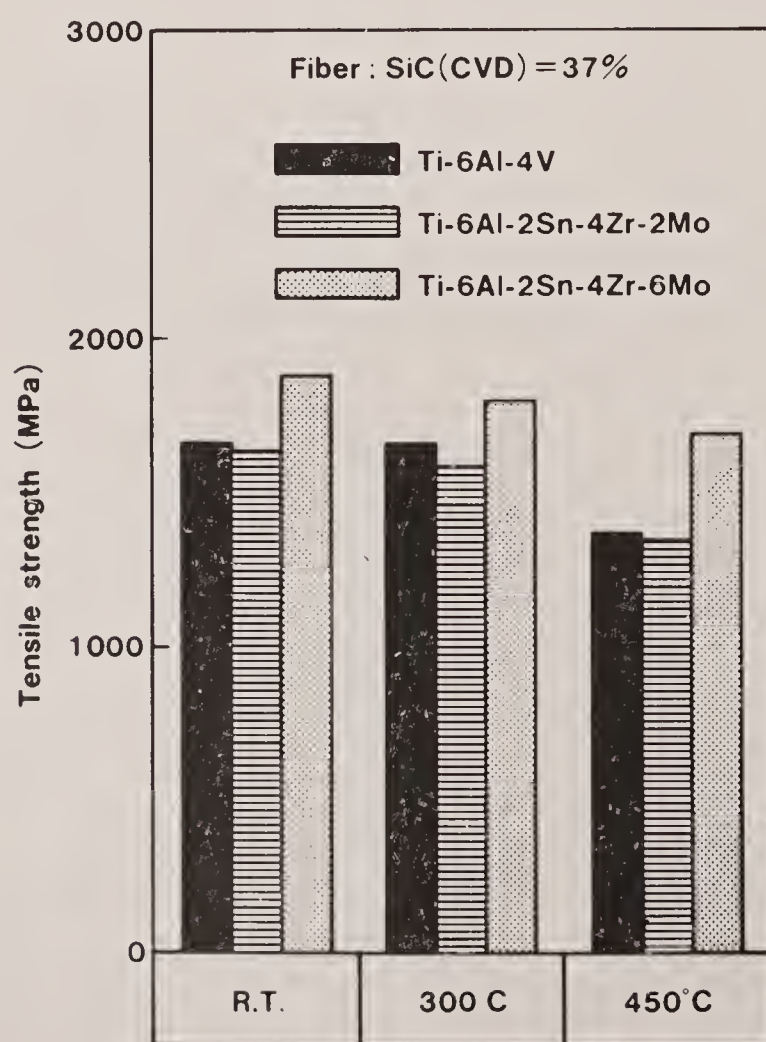


Figure 13. Effect of matrix on tensile strength of composites.

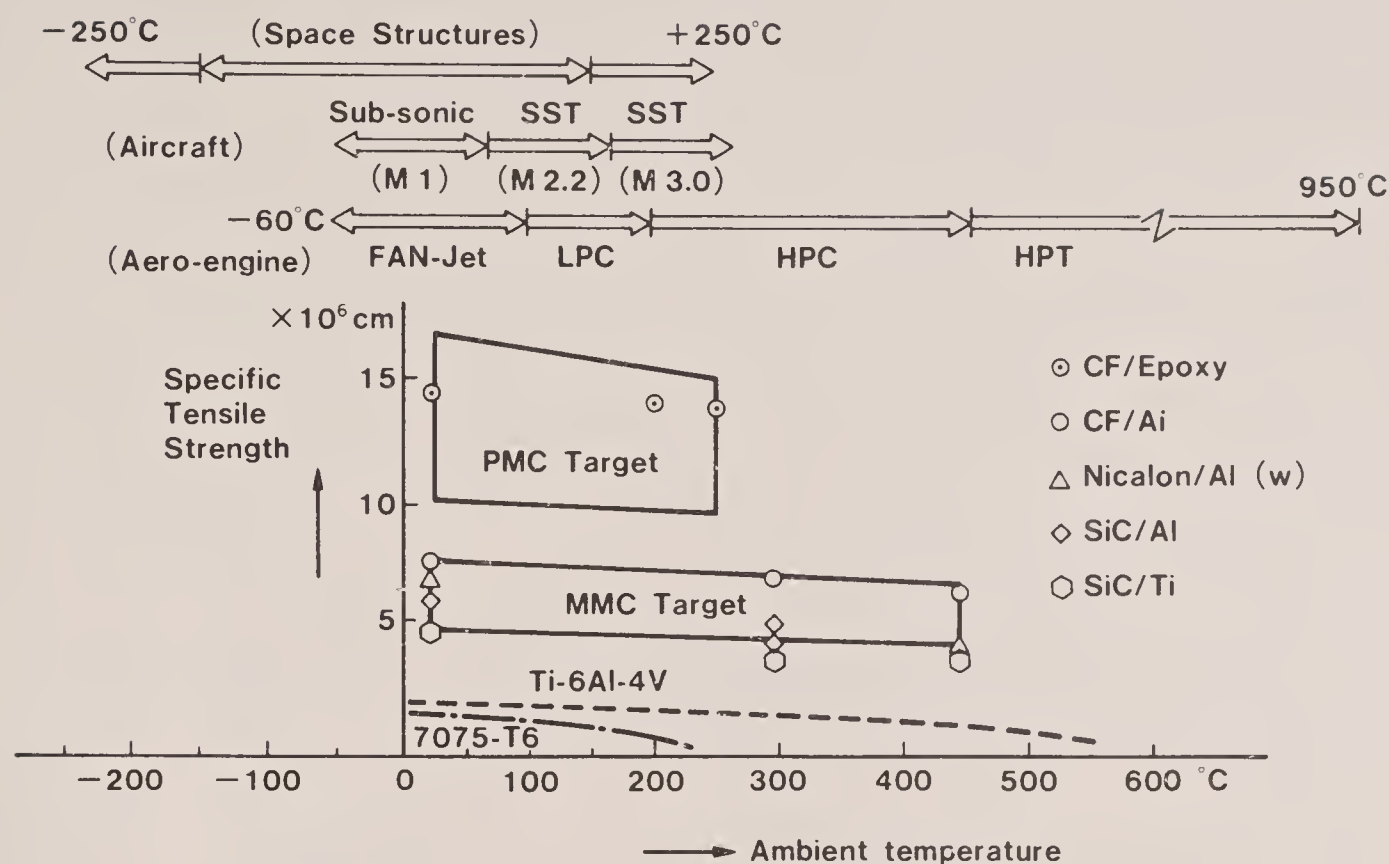


Figure 14. Attainment of target of the project for PMC and MMC.

4. Some structural applications of composite materials and trends in Japan

Recent FRP production and typical structural applications in Japan are outlined. The recent structural and non-structural application areas of composite materials have been expanding remarkably.

Some statistics of recent structural composite production in Japan are first introduced. Table 12 is the history of annual production market share of GFRP in our country for several years. The total amount of annual production has been gradually increasing. (Jpn. R P Soc. 1986)

Figure 15 shows the processing share of GFRP in which the hand-lay up process is still as high as 37%, but is gradually decreasing.

Table 13 is the corresponding data in USA. Figure 16 gives the consumptions of FRP in USA, Europe and Japan (SPI 1985).

Table 12. Production share of markets of GFRP in Japan.* ($\times 10^3$ ton).

Market	1982	1983	1984	1985	1986
Construction	24.8	23.2	24.9	25.4	25.5**
Housings	80.0	89.3	93.4	95.9	98.5
Marine	39.8	37.7	32.4	33.5	33.5
Transportation	4.9	4.9	7.3	8.7	14.0
Tank & vessel	32.0	33.2	35.0	35.7	36.8
Industrial goods	24.8	29.6	32.0	35.1	36.2
Miscellaneous	13.2	15.6	17.6	18.5	19.0
Others	13.4	15.2	14.2	15.4	15.5
Total	232.9	248.7	256.8	268.2	279.0

* Source: Data issued by Japan Reinforced Plastics Soc., 1986

** Estimated.

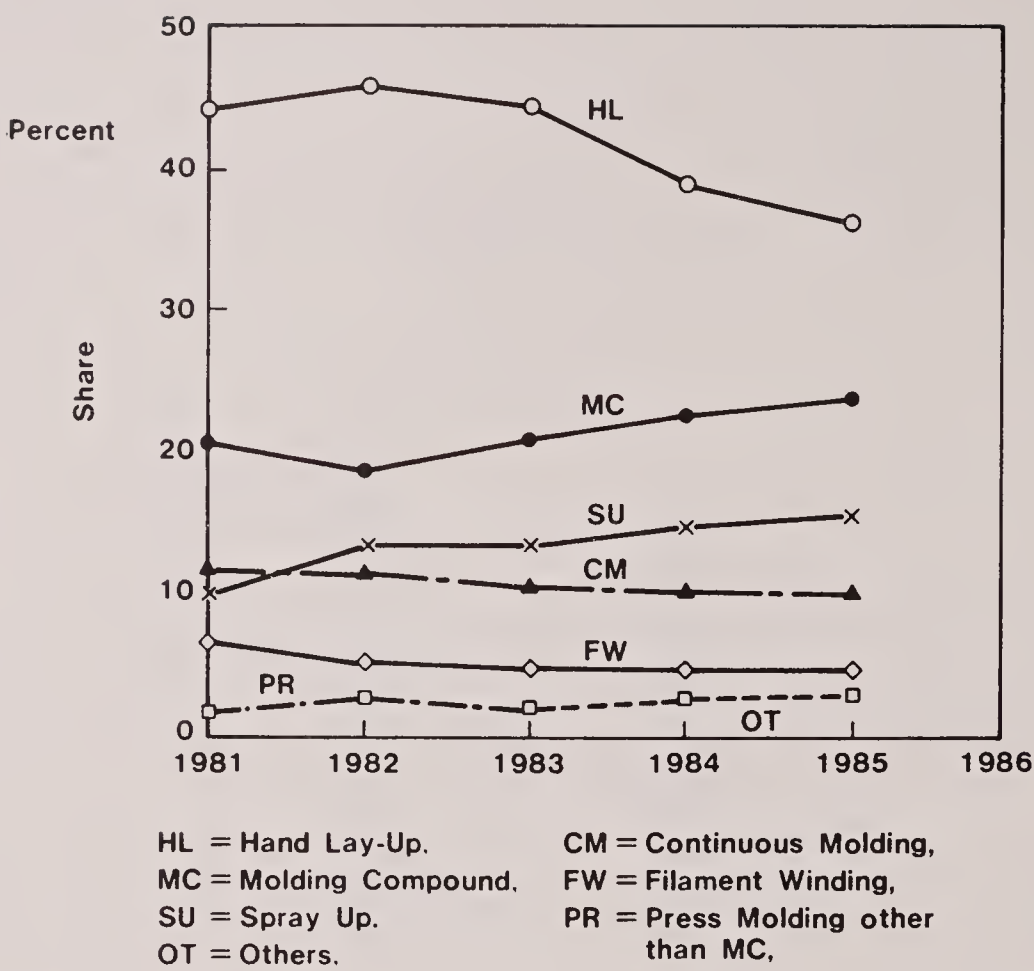


Figure 15. Share of processing for GFRP in Japan for years. (Data issued by Japan Reinforced Plastics Soc., 1985)

From these data, we can see the recent trend of FRP production and marketing in Japan, in comparison with those of USA and Europe.

In the following, some typical structural applications of composites are reviewed.

(1) STOL-“ASUKA” of National Aerospace Laboratory.

Principal characteristics:

Power plant: FJR710/600S – 4 engined,

Dimensions: Wing span = 30.6 m, Length = 29 m, Height = 10.245 m,

Wing area = 120.5 m², gross weight = 36.7 ton,

Performance: approach speed = 133 km/hr,

approach angle = – 6°,

take-off distance = 680 m (over 10.7 m high)

Table 13. Reinforced plastics shipments by markets in USA

Includes: reinforced thermoset and thermo-plastic resin composites

Includes: reinforcements and fibres (× 10³ ton.)

Market	1984	1985	1986
Aircraft	13.2(+ 16.0)	14.5(+ 10.3)	16.8(+ 15.0)
Appliances/business equipment	55.8(+ 16.0)	60.3(+ 8.1)	62.6(+ 4.0)
Construction	195.0(+ 8.0)	201.9(+ 3.5)	205.9(+ 1.9)
Consumer products	64.9(+ 12.0)	64.4(– 1.0)	65.8(+ 2.0)
Corrosion resistant equipment	140.6(+ 8.0)	133.8(– 4.8)	137.0(+ 2.4)
Electrical/electronic	85.7(+ 11.0)	86.6(+ 1.0)	90.3(+ 4.2)
Marine	140.2(+ 12.0)	152.0(+ 8.4)	157.4(+ 3.5)
Transportation	244.9(+ 18.0)	255.4(+ 4.2)	262.6(+ 2.8)
Specialties	36.3(+ 12.0)	37.2(+ 2.0)	38.6(+ 3.6)
Total	976.6(+ 12.0)	1006.1(+ 3.0)	1036.9(+ 3.1)

*Source: SPI Reinforced Plastics/Composites Institute (1985) and G.F. Grates *et al*: “Reinforced Plastics/Composites”, Industry has record year in 1985, 1986-shipments expected to continue pace.

Number in parentheses indicates annual rate of growth.

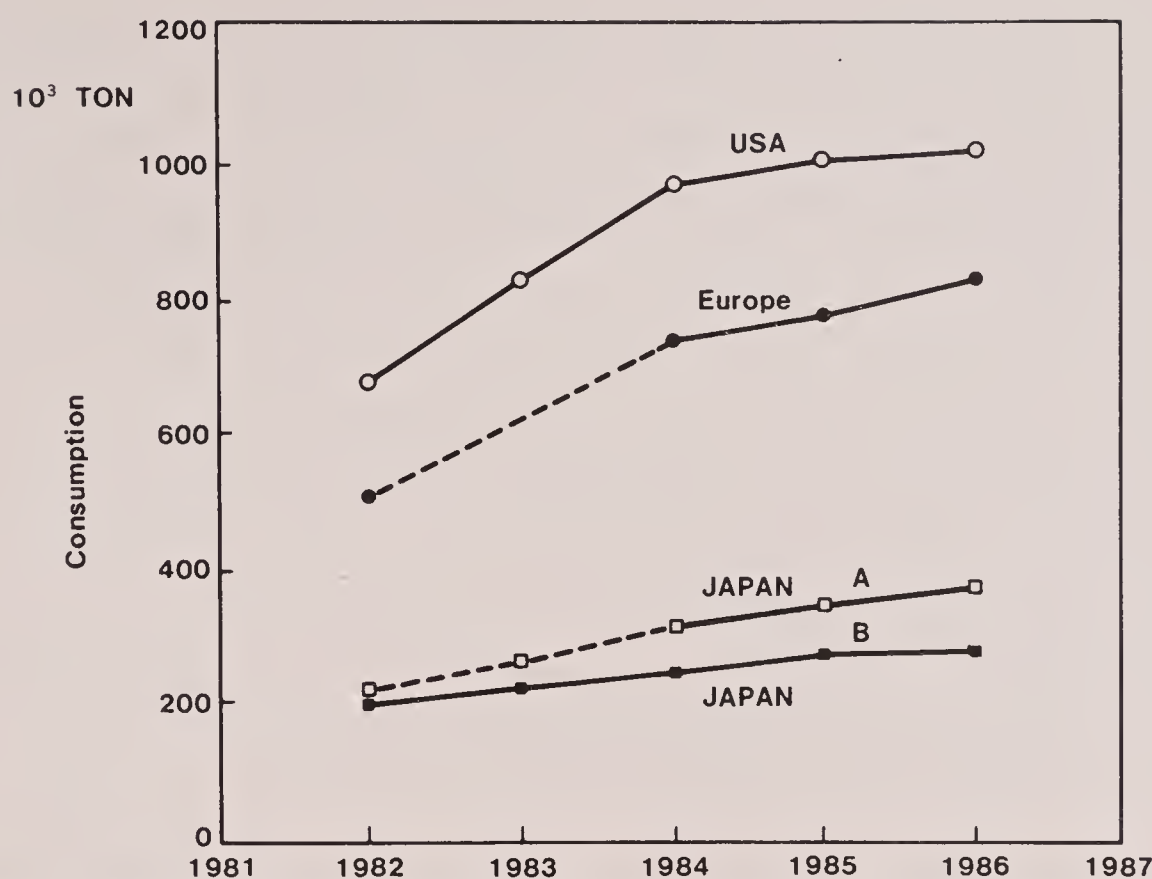


Figure 16. Consumption of polymeric composite materials in USA, Europe and Japan. USA = Polymeric composites, Western Europe = Polymeric composites for all common market countries plus Austria, Spain, Finland, Greece, Norway, Portugal, Sweden & Swiss. (Data from Modern Plastics International January 1986) JAPAN: A = GFRP + Other Composites, B = GFRP only. (Data issued by Japan Reinforced Plastics Soc., 1986, Data of 1986 is an estimation.)

landing distance = 480 m (over 10·7 m high)
 cruising speed = 0·565 Mach,
 range = 1,600 km.

High lift device = upper surface blowing (USB),

Heat resistant wing upper skin:

3 layers polyimide GFRP + Nomex honeycomb sandwich skin, Max. thermal load = 220°C.

- (2) Radio telescope antenna of the Astronomical Observatory of The University of Tokyo.

Total aperture = 45 m diameter,

Structure = CFRP-face plate and Al-honeycomb sandwich type structure,

Each panel size = 2·3 by 1·5 m,

Surface accuracy = 0·056 mm rms average,

Good resolving performance was attained.

- (3) Large superconducting thin solenoid magnet in National Laboratory for High Energy Physics.

TRISTAN-Project:

(MELCO)

7·5 kG magnetic field,

30 GeV e^+e^- collider,

length = 5^m 020 mm, outer diameter = 3^m 810 mm,

thickness = 27 mm, weight = 3 metric ton,

CF-prepreg tape = 150 mm width, 20 mm lap, winding angle = 2°, tape winding structure,

outgassing = 5×10^{-8} torr·l/s cm²,

Safety factor of FW cylinder for atmospheric pressure for buckling = 3.

- (4) Multi-joint manipulator robot.

Robot for inspection of critical environment where the multi-directionally movable manipulator, having high accuracy, high stiffness, light and good damping and stiff joints is required.

Arm speed: 5 m/s maximum. (TOSHIBA)

- (5) Curtain wall for building, made from glass fibre reinforced cement (GRC)

Some properties of a sample case:

GRC moulded by direct spray method:

$V_f = ca\ 3\%$,

Strengths: tensile = 1 ~ 1.5, compression = 8 ~ 10, interlaminar shear = 0.15 ~ 0.20, inplane shear = 1 ~ 1.5 kgf/mm².

Air dry specific gravity = 1.9 ~ 2.3,

Absolute specific gravity = 1.8 ~ 2.1,

Water absorption = 10 ~ 15%,

Thermal conductivity = 0.6 ~ 0.9 kcal/mhr°C.

Processing methods: premix, spray method, press etc.,

(ASAHI Glass Works)

(KASHIMA Building Co.)

- (6) Super-Jet Boat: KASUMI.

Hull structure = GFRP,

Dimension: length 28 m, width 5 m and depth 2.1 m,

average draught = 0.70 m, max. speed = 60 km/hr,

gross tonnage = 52 ton, cruising speed = 50 km/hr, number of passenger = 154, including 3 crew members,

engine = 700PS/2170RPM.

(YAMAHA)

- (7) Fibre reinforced polyvinylchloride (FRV) for off-shore structures.

$V_f = 18 \sim 23\%$, PVC = 77 ~ 82%,

Some properties of 1.5 mm thick FRV plate:

Strengths = tensile 6 ~ 14, compression 10 ~ 18, bending 13 ~ 28 kgf/mm²,

Specific gravity = 1.50 ~ 1.60,

Heat distortion temperature = 74 ~ 100 °C,

Water absorption = 0.04 ~ 0.05 wt%,

Good anti-corrosive property,

Good processability. (ASAHI Glass Works)

- (8) Pipes for waterworks.

For drainage, water supply and water power pipes with large diameters:

Outer diameter: 600 ~ 5200 mm,

Thickness: 12 ~ 60 mm,

Material: GF/polyester for outer layer,
resin/mortal for core layer.

- (9) Cooling water tanks.

For airconditioner placed on roof of building.

- (10) Wheeled chair for disabled persons.

CFRP pipe framework structure,

Total weight: 10 kgf.

(IPRI)

- (11) Linear motor car

Future dream car – non-polluting, high speed, suitable for mass transportation, energy saving and of low cost. Magnetic hover and linear motor drive vehicles, (A) HSST, (B) ML-500, (C) MLU001, 002 & 003.

(A) HSST (High speed surface transport)

Linear induction motor type vehicle.

HSST-no. 3 vehicle:

weight empty = 12 ton, gross weight = 15 ton,
dimensions: length = 13.7 m, width = 2.95 m, height = 3 m,
max. speed = 307.8 km/hr (1978),

Vehicle structure: Framework made of Al,
CFRP honeycomb panel flooring,

ceiling panels, window frames, door frames, console boxes were made of
GFRP-paper honeycomb sandwich panels covered with decorated paper.

design speed = max. 55 km/hr, normal 30 ~ 40 km/hr,
deceleration = normal 2.6 km/hr,

emergency 5.3 ~ 7.1 km/hr. (JAL)

(B) ML-500.

Linear synchronous motor type vehicle.

GFRP/Nomex honeycomb sandwich panel for canopy and cover, Gummi
tyres,

gross weight = 10 ton,

max. speed = 517 km/hr (1984). (JR)

(C) MLU-001.

Linear synchronous motor vehicle.

Repulsive hovering,

Linear synchronous motor vehicle.

thrust = 0–5.2 ton; 3000 V, 1100 A.

Dimensions: length = 30 m, width = 3 m and height = 3.3 m,

Gross weight = 10 ton:

share: body structure: 2.5 ton,

carriages: 6.5 ton (including superconducting magnet, its cryostat, carriage
frame and accessories).

others: 1.0 ton,

Materials: FRP/Al-honeycomb,

FRP/urethane foam sandwiches. (JR)

(12) Space structures.

Satellite structure, antenna, fairing, rocket.

There are many other applications in our country, but only a limited number of
applications are listed above, listing some of their features.

Composite materials are now expanding their application areas not only to
secondary structures of aero-space, automobile and other transport vehicles, but also to
primary structures. On the other hand, non-structural applications are now increasing,
for example, in electronics and medical fields and so on.

5. Current problems and future trends

5.1 Establishment of composite material science and technology

In the field of composites, miscellaneous advances have been made in the past few
decades. Study of their science and technology, however, is still incomplete. Among
these are the following problems.

- (1) Establishment of theory, concerning fracture (static and dynamic), environmental degradation, fatigue and creep, slurry of reinforcement and matrix inside the mould under temperature and pressure gradients, polymerization accompanying flow, performance of multi-phase hybrid composites and functional composites have been the current areas of fundamental research.
- (2) In the next decade, composites resistant to much higher temperatures would be required. For instance, the thermal stabilities of polymer matrix composites (PMC), metal matrix composites (MMC) and ceramics matrix composites (CMC) up to 400°C, 650°C and 1200°C, respectively, are already quite promising. In particular, CMC are very hopeful candidates because of their high potential for thermal stability.
- (3) Computational mechanics, simulation technology, CAD and CAD/CAM are indispensable tools for the development of composite materials and structures. Rapid progress in these at present is essential.
- (4) Thermo-plastics which can be die-pressed, inter-penetrating network systems and molecular design of composites are also showing some progress.
- (5) Enhancement of reliability of fabrication technology.
- (6) Rational application of composites in structural designs.
- (7) Multi-phase composites are newly emerging areas of research.
- (8) Submicron or even thinner films of reinforcement and matrix would be required for electronic semi-conductor fields.

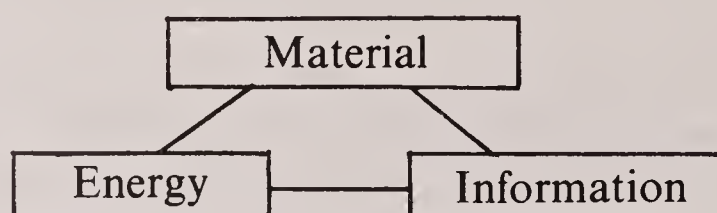
5.2 Data bank problem

Before data acquisition, work on composites evaluation technology and standardization of test methods are urgently needed. Comprehensive and systematic data acquisition should then follow.

For this work, it would be necessary to promote international cooperation.

5.3 Leading philosophy and basic issues for future industry

- (1) Three basic issues for future industry: It is widely recognized that the strong driving and leading source for future industry will be the following three basic issues, (A) materials, (B) energy and (C) information. These would form the core for future studies mutually interacting and influencing each other.



- (2) How to evolve the philosophy and strategy for future industry.

The philosophy of past industrial work has been the forcing of structural changes toward the next decade. Responding to the needs of the era, what should we do for future industry in our country? Some views are given in table 14.

This table should be read as follows. For example, for “production”, the current conventional ideas: “few models and mass production” should be changed to “diversified models and minor production” and so on. This modified philosophy would be reflected in our future industry.

Table 14. Modification of philosophy in the next decades

Factor	Era of industrial production From	Era of infor- mation To
Consumer needs	Uniform	Diversified
Technology	Dependent	Self development
Production	Few models mass production	Diverse models minor pro- duction
Efficiency	One side efficiency	Global & harmonized efficiency
View of worth	Emphasis on efficiency only	Emphasis on originality
Quality	Labour-intensive	Knowledge-intensive
Design principle	Heavy, thick, long, big	Light, thin, short, tiny
Capital	Small	Large
System	Pyramid type	Network type
Decision making	Top	Mutual cooperation
Flow of information	From top to bottom	Multi-cellular multi-path way
Input & output of strategy	Low risk & high return	High risk & high return

6. Conclusion

Brief results of AIST R & D project for composite materials since 1981 to the present were discussed. Several innovative developments and various problem areas were pointed out.

In order to judge the utility of composites, it is useful to visualise the areas of application. Hence some structural application areas were also introduced.

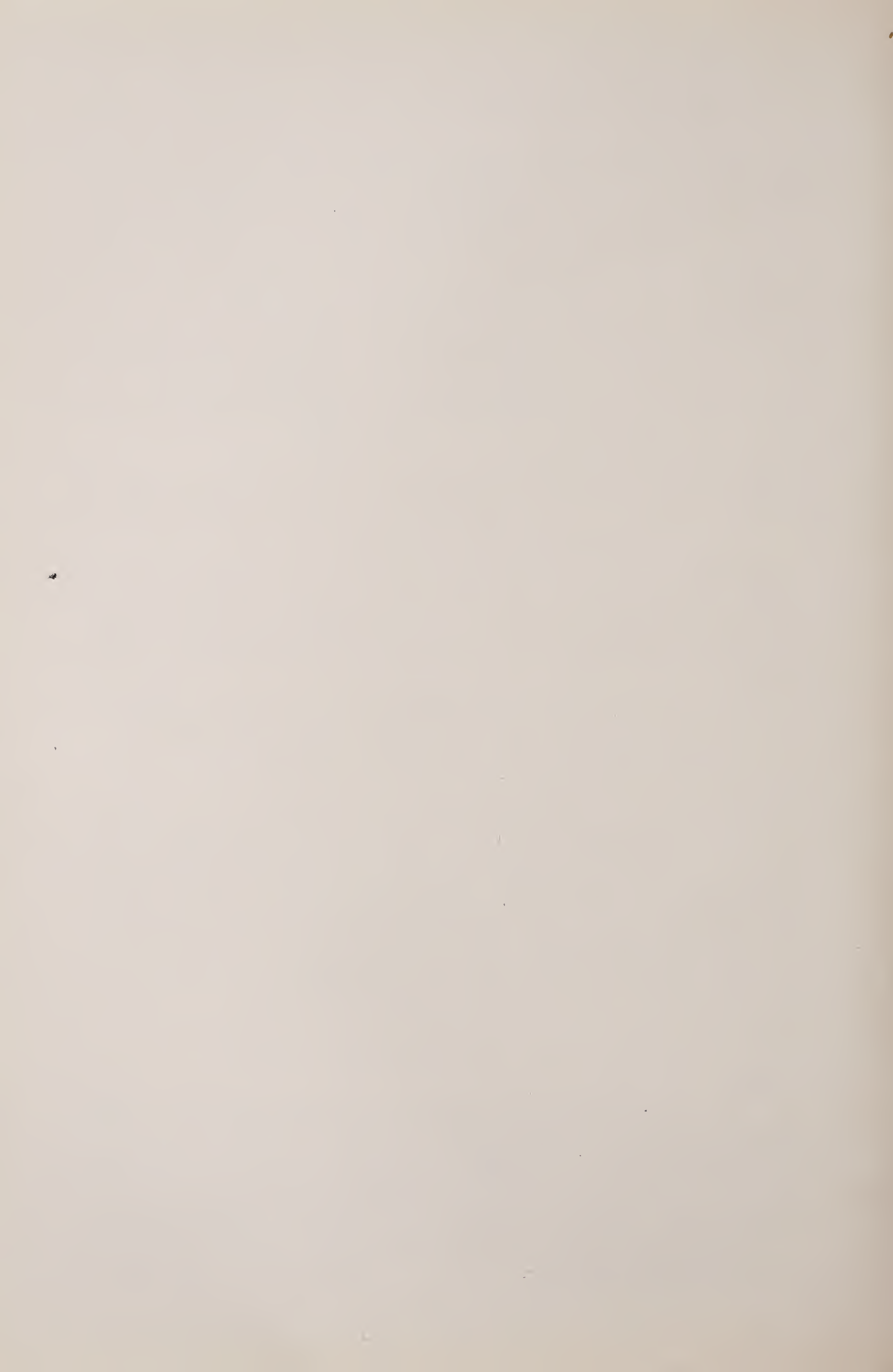
Current problems and future trends were considered, together with possible changes and modifications of concepts in future industry in our country.

Dr R C Horney, DuPont Co., has estimated that the worldwide market for composites will be about \$23 billion by the late 1990's, with a share of 13% in basic components and prepregs, and about 87% in finished parts of composites (Horney 1985). Dr. Horney has also suggested that by the turn of the century the share of polymeric composites will reach 20% of the total dollar volume of structural material and almost 50% of it by the year 2030 (Horney 1985).

The author would like to conclude suggesting that there may exist very wide areas of application of composites by extension of the present solid phase composites to the liquid phase, and further to the solid-liquid hybrid phase, where bio-materials may well be included.

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R & D work in composites at the Fibre-Reinforced Plastics Research Centre

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Abstract. This paper briefly presents some of the R & D activities taken up by the Fibre-Reinforced Plastics (FRP) Research Centre, Indian Institute of Technology, Madras, in the last seven years. The studies covered in this paper include the analysis of heat shield, development of vibration mounting for rocket motor casings, design and development of metal-lined pressure bottles, study of the effect of nuclear radiation on filament wound shells, design and development of high voltage disc insulators, study of underwater-acoustic characteristics of FRP and development of some of the processing techniques. The paper also lists a number of products developed by the Centre for meeting the immediate needs of society. Finally, the on-going academic and sponsored research programmes are briefly outlined.

Keywords. Composites; fibre-reinforced plastics.

1. Introduction

The Fibre-Reinforced Plastics (FRP) Research Centre was set up by the Indian Institute of Technology (IIT), Madras, in 1974, with a view to take up research, teaching, technology development and consultancy in the area of composites technology. It is one of the four interdisciplinary research centres of the Institute and has established over the last 13 years an identity of its own, and has good rapport with industries and other institutions in the country.

The research and development activities of the Centre can be broadly grouped into the following:

- (i) academic research aimed at generating scientific knowledge;
- (ii) R & D studies to stimulate the use of composites for certain high technology developments in the country;
- (iii) The development of certain products that directly benefit society.

Six papers (Babu 1988; Kari Thangaratnam *et al* 1988; Malhotra *et al* 1988; Nair & Premachandran 1988; Rajanna *et al* 1988; Santhanakrishnan & Krishnamoorthy 1988) being presented at the International Conference on Composite Materials and Structures (ICCMS-88) at IIT, Madras, reflect the nature of the academic research

activities at the Centre. They include studies in the area of micromechanics, rheology of composites, buckling, flutter and dynamic analyses of laminated composites using finite element methods, software development for analyses of composite structures, and machining and friction characteristics of fibrous composites.

The development and use of technology requires certain studies that are of immediate interest to the nation. Although a developing country like India with R & D activities in several high technology fields needs the use of composite materials, it is also necessary to study and find out how best composite materials can be put to use for general social development. This paper briefly summarizes some of the R & D studies taken up by the FRP Centre towards these goals.

2. Background

Industrial activity in the area of composite materials started in the country in 1962 when Fibreglass Pilkington Ltd. was established for producing glass fibre in India. Research activities in the field of composites also started in the early 60s, but the activities during the 60s and early 70s were mainly confined to the theoretical studies on composite structures, treating them as anisotropic laminates or sandwich materials. The National Aeronautical Laboratory (NAL), Bangalore, and the FRP division of the Vikram Sarabhai Space Centre, Trivandrum, were the two organizations engaged in the development of products and processing technology in composites during this early period. In the early 1970s, the National Committee for Science and Technology of the Government of India emphasised that the development of composite materials must be taken up as a thrust area and importance must be given both for research and for manpower development in this field. In order to effectively take part in this national effort, IIT, Madras, set up the FRP Research Centre with the following objectives:

- to work for the advancement of composites technology and its applications;
- to undertake teaching, research, manpower training and other technical activities in order to meet the requirements of the FRP and allied industries;
- to provide advice, consultancy and technical leadership to the FRP and other composite materials industry in the country.

While theoretical studies have been given due importance, equal emphasis has been placed on experimental investigations, product development, design consultancy, and testing and quality control guidance to the industry so that IIT, Madras, can effectively contribute to the development of composites technology in the country. The Centre has now 5 faculty members and 9 supporting staff and is equipped with a composite materials laboratory, composite structures laboratory, FRP workshop and machine shop. A composites facility funded by the Department of Science & Technology, Government of India, New Delhi, exists at the Centre for providing testing and quality control services to the industries concerned.

3. Analysis of re-entry vehicles

Re-entry vehicles are used in space flights and missiles technology for bringing back a payload from space into the earth's atmosphere. The payload may include astronauts,

sophisticated instruments, technical data collected by the satellite during its space flight or some warhead in the case of missiles. The vehicle that enters the earth's atmosphere at an altitude of about 80 km above sea level at velocities of the order of Mach number 11 at re-entry, experiences aerodynamic heating raising the temperature around the vehicle to about 2600 K. A heat shield made of carbon-phenolic or carbon-carbon is used for dissipating the heat by the ablation of the material and for protecting the payload within allowable limits of the inside temperature (300 K). A study was conducted at the Centre by Mohideen (1985) for developing the analysis and design capability for predicting the trajectory, and determination of skin temperature on the heat shield, temperature distribution through the thickness of the heat shield, and the stresses developed in the heat shield due to the aerodynamic pressure loading as well as due to the thermal gradient through the thickness. Figure 1a shows the shape and dimension of a heat shield chosen for the analysis. Stress analysis techniques have been developed using finite element methods for the following cases:

- (i) Stress analysis under the axisymmetric loading of aerodynamic pressure, deceleration and thermal stresses. SAPIV four-node isoparametric 2D and beam elements have been used for the analysis. Figures 1b and c show axial and circumferential stresses plotted along the length due to the combined effect of pressure, inertial and thermal loadings.
- (ii) Stress analysis under non-axisymmetric loading corresponding to the non-zero angle of attack. Shell elements have been used for the finite element analysis.
- (iii) Stress analyses of the regions of high stresses using 3D elements.
- (iv) Buckling analyses of the shells under external pressure and thermal loading using semi-loof shell elements specially developed for composite materials.
- (v) Frequency and mode shape analysis of the shell by the finite element method.

The FRP Research Centre has developed the competence and capability to carry out the design and analysis of heat shields of re-entry vehicles.

4. Vibration mountings of rocket-motor casing

The vibration characteristics of rocket-motor casings are studied by mounting them on conical shell supports of the type shown in figure 2. These shells must be light and stiff and the mounting under design must have natural frequencies in the range 800–1000 Hz so that they will not interfere with the vibration of the motor casings. Traditionally, magnesium alloys are made use of in making these mountings. Fabrication of magnesium alloy mounting shells involve costly machining operations and such mountings are currently being imported.

A study was taken up at the FRP Research Centre to see whether FRP could be used for fabricating such mountings. Carbon fibre-epoxy composites, although a potential materials system, were not considered due to their high cost. Instead, glass-epoxy shells with appropriate stiffening webs in-laid with aluminium were chosen for the study. The dimensions of the shell wall, stiffener size and thickness, dimensions of the aluminium inserts etc. were determined by a finite element analysis. The designed mounting was then fabricated by the hand lay-up method. Extensive evaluation of the mounting was carried out by the SHAR Centre of The Indian Space Research Organization and this study has shown that the natural frequency achieved was less than the value predicted by the finite element method of analysis. The shell was further

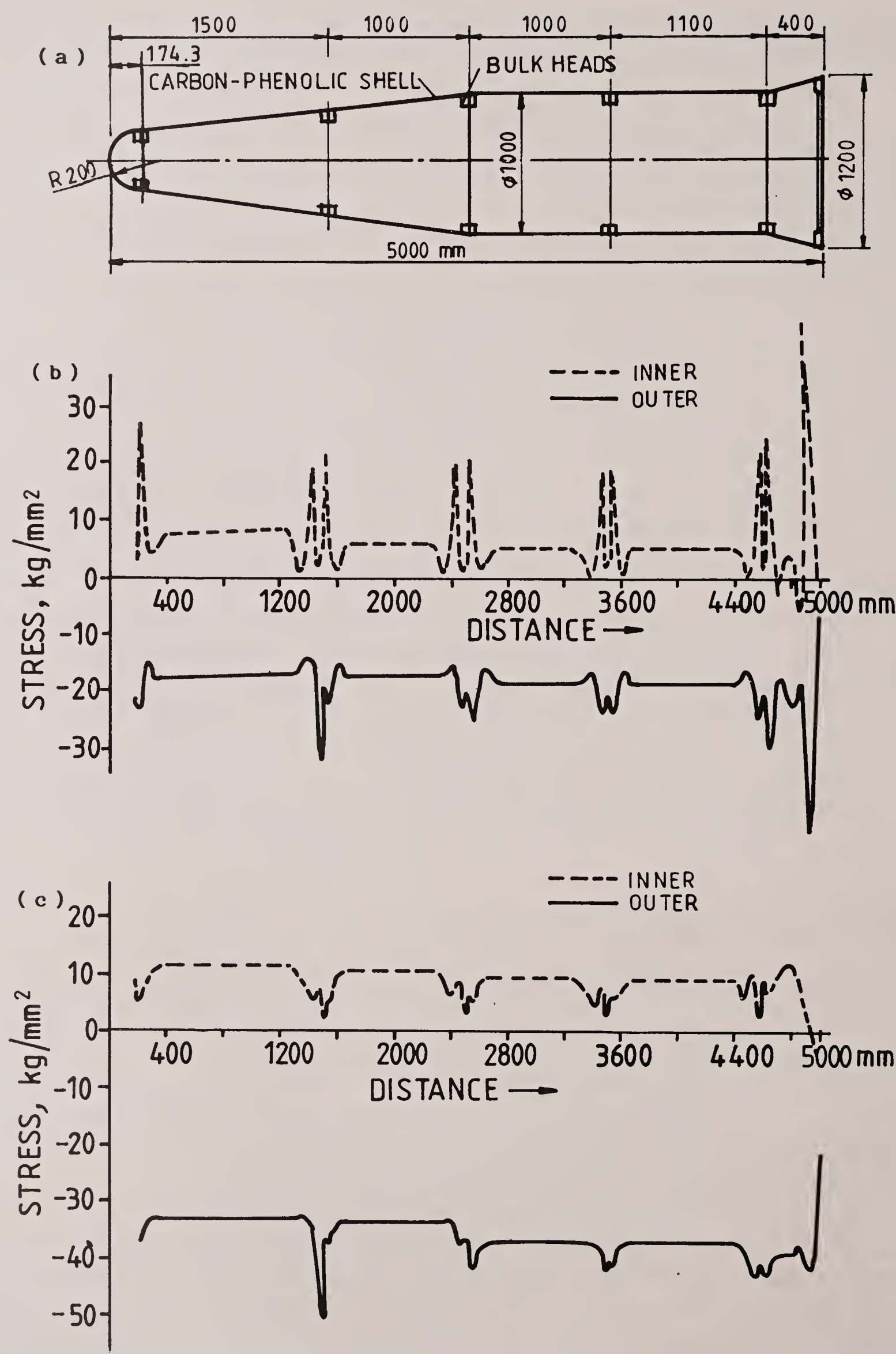


Figure 1. Stress distribution along the heat shield due to aerodynamic pressure; inertia and thermal loading. (a) Assumed shape of heat shield; (b) axial stress, and (c) circumferential stress.

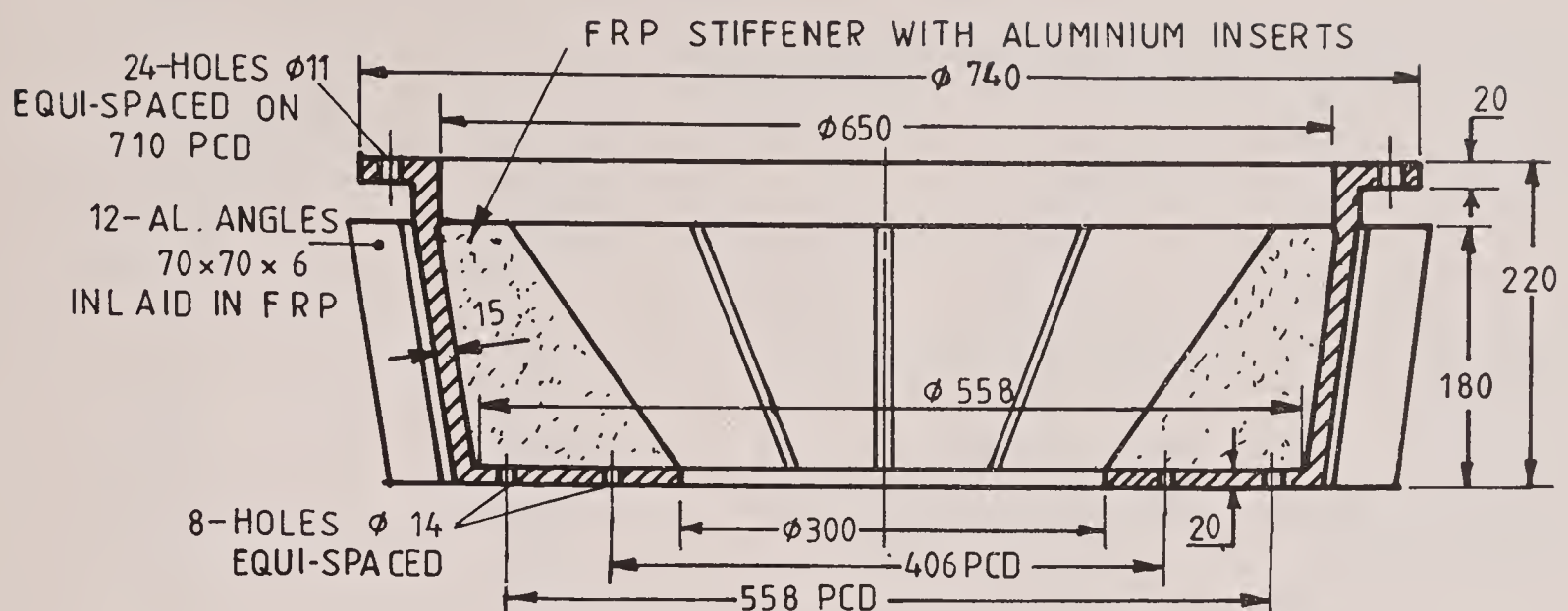


Figure 2. FRP vibration mounting.

stiffened by adding more stiffeners and glassfibre-reinforced plastic (GRP) layers. The improved vibration mounting is now undergoing trials at the SHAR Centre.

5. Metal-lined FRP pressure bottles

FRP pressure bottles find many applications in aerospace, hydrospace and military fields. They are used primarily for storing gases or liquified gases under high pressures. Applications include oxygen bottles, underwater buoys, missile casings, gas bottles for guidance of spacecraft etc. Pressure bottles made by the filament winding of glass, Kevlar or carbon fibre-reinforced epoxy composites develop matrix cracking at relatively low stress levels. Therefore, they are internally lined with metallic or elastomeric liners which provide impermeability even when the matrix has cracked and which allow the bottle to be used at higher pressure levels without weeping of the gases. Unlike elastomeric liners, metallic liners, if bonded properly, act as integral parts of the FRP shells. The metallic liner has the added advantage that it serves as a mandrel for polar winding of the pressure bottle.

Under a sponsored programme (Malhotra & Babu 1985), the FRP Research Centre took up a project for the design and fabrication of metal lined pressure bottles. The pressures to be withstood are upto 400 atmospheres. Glass epoxy was chosen as the material for fabrication of the shell because of its cost-effectiveness over carbon or Kevlar-epoxy systems.

5.1 Development

A study of the bonding of aluminium and stainless steel with various types of GRP by a series of lap shear tests (listed in table 1) has shown that aluminium and stainless steel with etching have good bonding with the glass-epoxy material. Etching has been done with a mixture of potassium dichromate, sulphuric acid and distilled water at 65–70° C. Aluminium is also compatible with filament wound glass-epoxy because its modulus is closer to that of the GRP than that of stainless steel. Because of this compatibility and because of its light weight and production advantage, aluminium was chosen as the liner material. Figure 3 shows the shape and dimensions of a typical aluminium liner with the filament wound FRP over the liner.

Table 1. Lap shear strength of metal-FRP bonded interface.

Metal layer used	FRP structure	Lap shear strength (N/mm ²)	
		Without etching of metal	With etching of metal
Aluminium	Glass CSM* and polyester	2·12	7·81
Aluminium	Glass WRM† and polyester	2·07	6·73
Aluminium	Glass WRM and epoxy	3·63	10·13
Stainless steel	Glassfibre CSM and polyester	2·28	4·86
Stainless steel	Glass WRM and polyester	4·74	16·09
Stainless steel	Glass WRM and epoxy	5·32	15·50

* Chopped-strand mat (source: Malhotra & Babu 1985);
† Woven roving-mat.

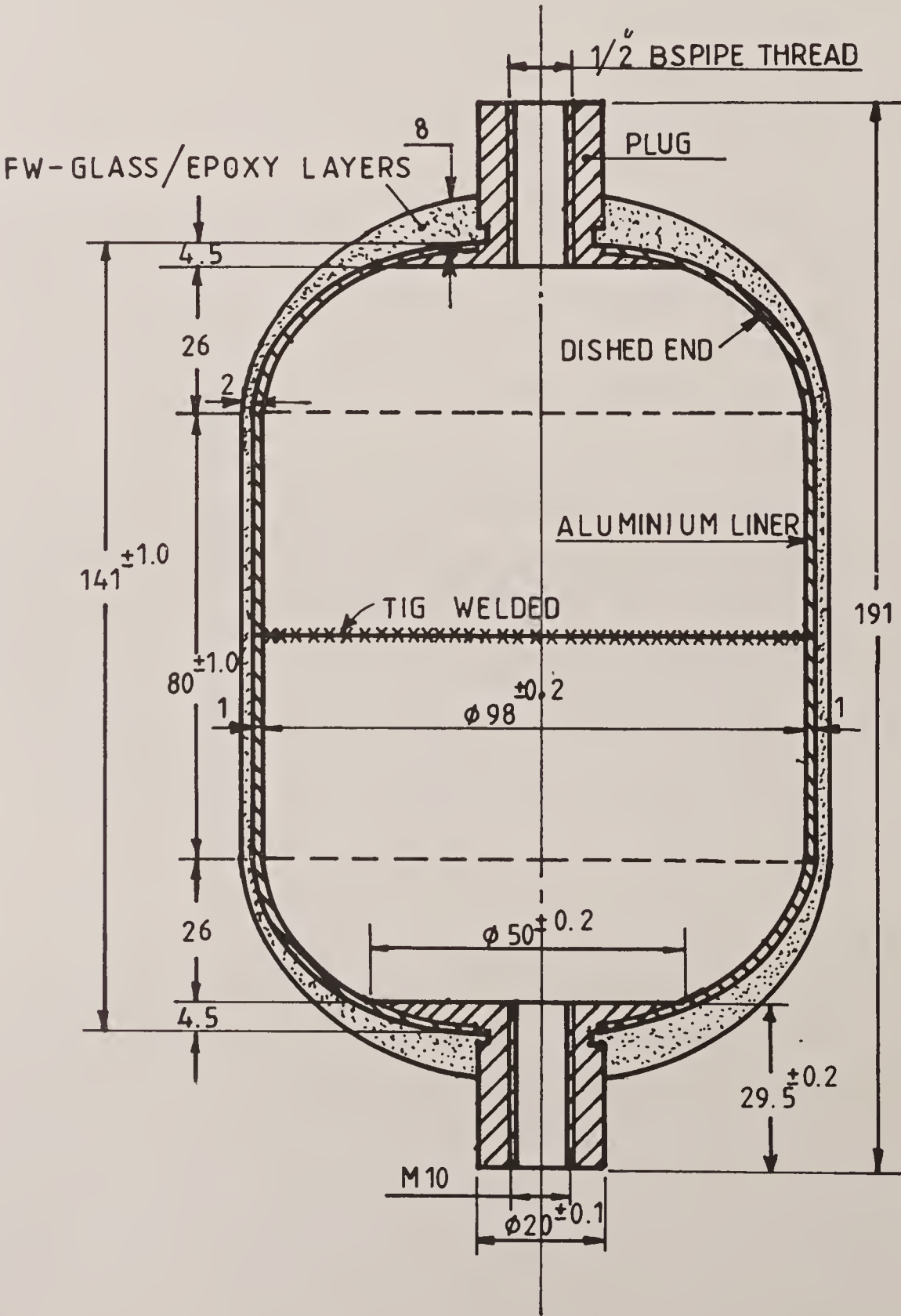


Figure 3 Glass-epoxy pressure bottles.

Table 2. Burst strength of metal-lined FRP pressure bottles.

Thickness of aluminium liner (mm)	FRP shell structure			Burst pressure (kg/cm ²)
	Roving tex	No. of double layers of polar winding	No. of layers of hoop winding	
1	Nil	Nil	Nil	6
Nil	1140	1	2	75
1	1140	1	2	165
1	1140	2	2	290
1	450	2	2	140
1	450	3	2	195
1	450	4	2	255

(Source: Malhotra & Babu 1985)

5.2 Analysis

A special purpose finite element analysis package using a four-node shell element with four degrees of freedom per node was developed for analysing the pressure bottle. The relative dimensions of the liner and FRP shell and the thickness of the FRP shell for strain control are decided based on this FEM analysis. An FEM can be used to predict the stresses and strains until the liner plastically yields or until the resin cracks, whichever is earlier.

5.3 Fabrication and testing

Pressure bottles were fabricated by using polar winding machine-designed and manufactured at the Centre. Glass-epoxy prepreg rovings were made in a laboratory model prepreg making set-up which is available at the Centre. The prepreg rovings were then wound on the liner, after which the bottles were cured in an oven. Pressure bottles with different layer thicknesses were made and tested in a burst pressure test set up. Table 2 gives the various ultimate pressures obtained in these cases. Design and fabrication of pressure bottles of up to 500 mm diameter that can withstand pressures up to 400 atmospheres is now possible.

6. Effect of nuclear radiation on filament wound FRP pipes

A project (Malhotra & Radhakrishnan 1984) was taken up to study the suitability of GRP pipes for use in nuclear radiation environment. Under this programme, glass-epoxy and glass-polyester-filament-wound pipes were fabricated and their properties determined before exposing them to nuclear radiation. The tubes were then subjected to five different total doses of nuclear radiation and then tested for their mechanical properties. Table 3 shows the percentage reduction in the axial tensile strength, axial compressive strength and hoop stiffness for various doses of nuclear radiation. The study has revealed that glass-polyester material is not suitable for long-term exposure to nuclear radiation whereas glass-epoxy pipes can be used for not-so-critical piping systems in nuclear plants. The very high reduction in hoop stiffness as compared to the

Table 3. Reduction in mechanical properties of filament wound FRP pipe due to nuclear radiation.

Total dose of radiation (mega rads)	Reduction in mechanical properties (%)		
	Axial tensile strength	Axial compressive strength	Diametral stiffness EI
Glassfibre–epoxy pipes			
244·12	6·60	4·25	20·00
298·91	6·38	6·25	24·00
655·58	6·86	7·13	22·39
973·55	7·24	7·82	23·4
1533·46	8·09	8·53	22·7
Glassfibre–polyester pipes			
244·12	9·30	4·96	7·09
298·91	9·63	11·78	12·75
973·55	12·79	13·93	14·73

(Source: Malhotra & Radhakrishnan 1984)

reduction in axial strength shows that nuclear radiation affects plastics more than it affects glass fibre.

7. Underwater acoustic performance of GRP

Underwater surveillance is generally based on sound signals because of the very low attenuation that sound waves suffer in the saline water of the oceans. In addition to substances like the piezo-electric materials used for electro-acoustic transduction, a few other materials, generally called passive materials, are being used as sound absorbers, sound reflectors or acoustic window materials. Glassfibre-reinforced plastics find application in sonar domes as acoustic window materials because of the combination of high mechanical properties, corrosion resistance and transparency to sonar signals offered by this material.

A study was taken up as a part of the academic programme (Narayana Das 1987) to understand the transmission and reflection characteristics of GRP and the effect of various materials parameters on these acoustic properties. GRP laminates of 0·5 m × 0·5 m were prepared using glass fibre in chopped-strand mat and woven roving-mat forms. Laminates with different layer thicknesses, fibre contents and void contents were prepared. Acoustic properties were measured in an acoustic tank facility. The study has yielded a number of behavioural characteristics of GRP. As an example, figure 4 shows the variation of maximum transmission loss with fibre content and void content for a woven roving-mat and chopped-strand mat laminate. A theory has been formulated for predicting the transmission characteristics of multi-layered FRP laminates using the experimentally obtained properties of the lamina.

8. FRP insulators for HV transmission lines

Ceramics are traditionally used for making disc/string insulators for the overhead

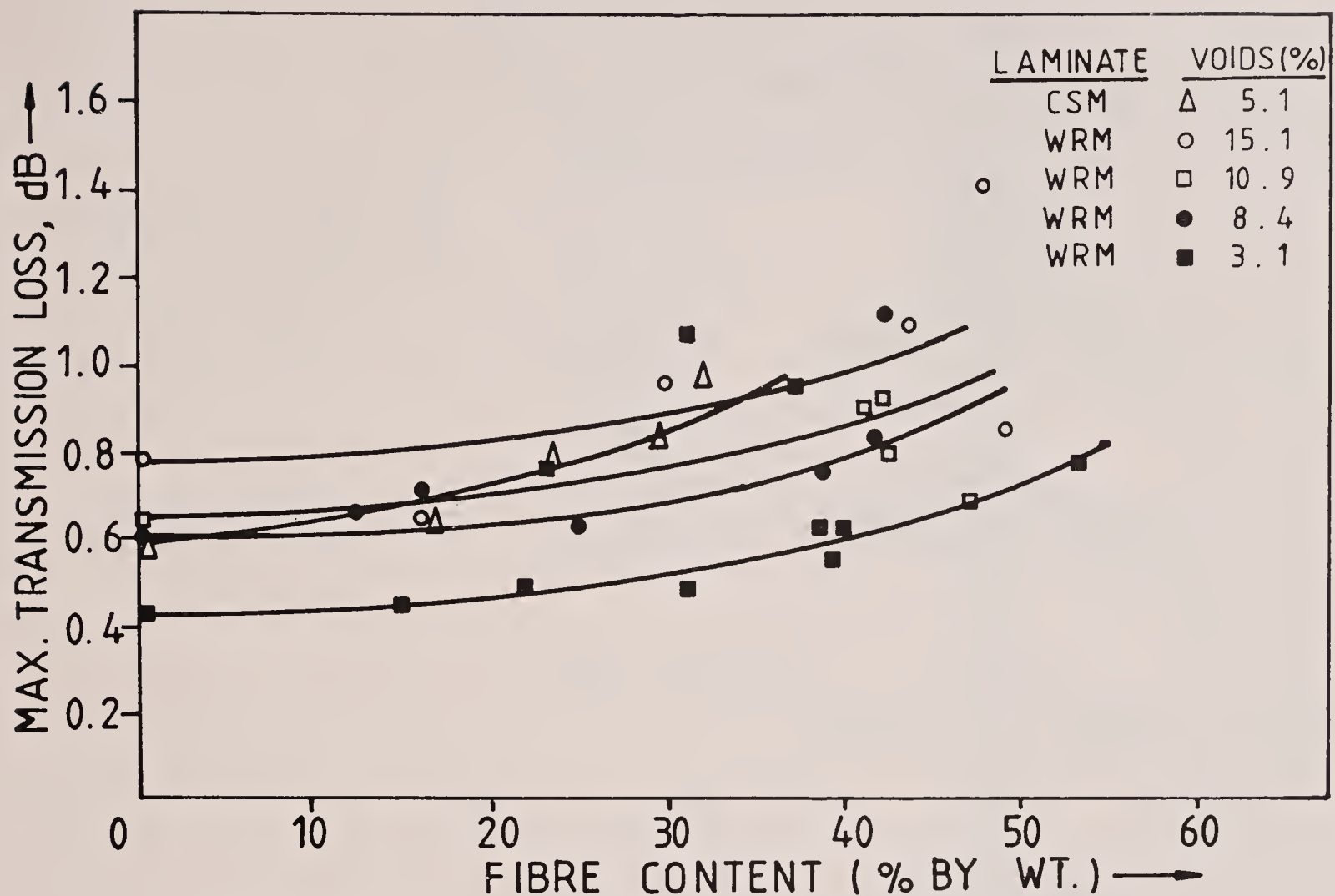


Figure 4 Effect of microstructure of FRP on the acoustic transmission loss.

transmission lines. They have excellent insulation properties and durability, but have poor impact strength and are heavy. The FRP Research Centre, under a master's degree programme for entrepreneurship, has developed a composite insulator system for high voltage (HV) transmission lines and this design can be easily extended to extra high voltage (EHV) lines. A multi-disc string insulator can be made either by assembling single-disc insulators as in the conventional system or by casting the discs over a central FRP rod. The weight of a five-disc insulator is less than 40% of the weight of an equivalent ceramic insulator. The discs have been designed to satisfy Indian Standard specifications. The product has been tested at the High Voltage Laboratory of IIT, Madras, and has been found to meet all the requirements as per Indian Standards. Figure 5 shows the typical section of a five-disc insulator cast on an FRP rod.

9. Development of process technology

Growth in the application of composite materials can be accelerated only if various processing technologies are developed indigenously. In order to achieve this goal, several process development studies have been taken up at the Centre. (Premachandra 1984) has studied the rheological characteristics of injection moulding of reinforced thermoplastics using coaxially extruded glassfibre-reinforced thermoplastic pellets. The results of this study are quite useful for injection moulding of glass-filled materials in ram-type injection-moulding machines. A Rajanna (unpublished) has developed the reinforced reaction injection moulding (RRIM) of polymethyl methacrylate (PMMA). The fibre is packed in a closed mould and a reaction mixture of MMA is injected into the mould. The process yields a component in $2\frac{1}{2}$ hr as compared to the 6 hr required in conventional PMMA coating. Pradyut Datta (1984) has developed the process of coating

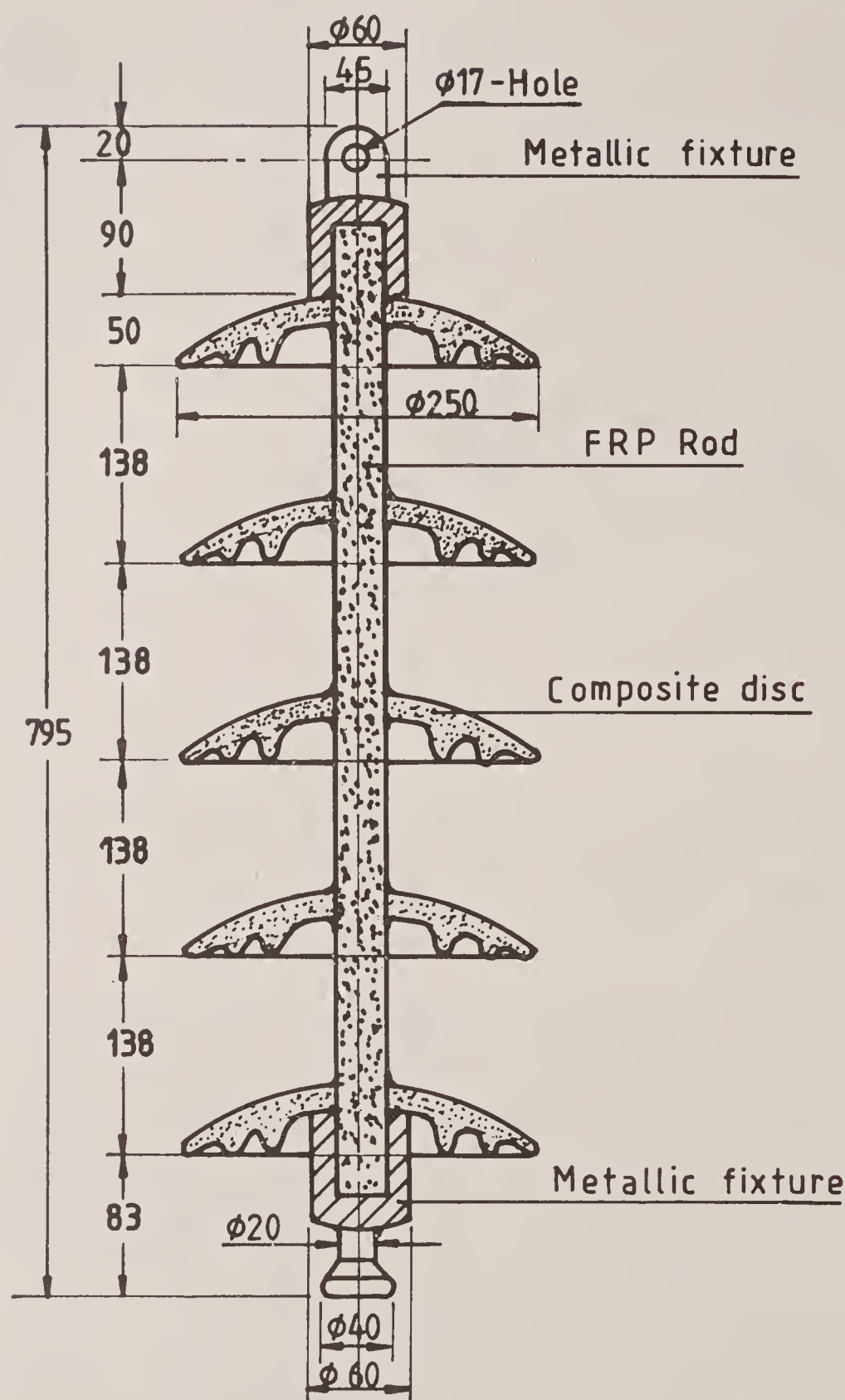


Figure 5. Composite multi-disc string insulators.

PVC, PTFE and polyurethane over glassfibre woven roving-mats and woven yarn cloth. These coated fabrics have high strength and impermeability making them suitable for inflated structures and biogas holders. Desai (1986) has developed a polymer-concrete-processing technique both for the surface finish grade and structural grade.

10. Product development

The adaptation of technology to meet immediate social needs cannot be ignored in any research programme taken up particularly by a developing country. FRP can be effectively utilized for a number of products that may directly benefit society or industrial production. A few products of this type as listed in table 4 have been developed by the FRP Research Centre.

India has an abundant supply of natural organic fibres like jute, coir, pineapple fibre etc. Among these fibres, jute has been found to have excellent mechanical properties. Under a sponsored programme, studies have been conducted to develop jute-polyester

Table 4. List of FRP products developed and fabricated at the Centre.

Product	Material used	Processing method
Prefabricated house (20 m ² area)	Jute-polyester	Hand lay-up
Prefabricated house (20 m ² area)	Jute-polyester	Filament winding
Window frames of express buses	Glass-polyester	Squeeze moulding
Doors of express buses	Glass-polyester	Hand lay-up
Tubes for H.V. Insulation	Glass-epoxy	Filament winding
Radio frequency former	Glass-epoxy	Filament winding and machining
Bio-gas holder	Glass-polyester	Hand lay-up
Microwave antenna (0.5, 1.2, 3.3 m dia.)	Glass-polyester	Hand lay-up
Wear-resistant composites	Silicon carbide-epoxy	Vibration compaction
Polymer concrete products	Glass-polyester	Vibration compaction and compression moulding

composites and to develop products like houses, silos and fishing boats. Jute-polyester laminates and sandwich sections have been developed and their load carrying capacity has been evaluated. The weathering of jute fibre and the consequent reduction in mechanical properties have been determined over a period of one year by exposing them to the natural environment, burying them under ground, and immersing them in fresh/sea water. Jute-polyester composites have shown excellent load-bearing characteristics but a gradual degradation of mechanical properties with time has also been observed.

11. On-going research programmes

A few research programmes initiated recently include theoretical studies, design and process technology development in different areas. Some of them are given below:

- (i) delamination of laminated composites is being studied both theoretically using FEM techniques and experimentally using flaw detectors;
- (ii) a computer-aided design system is being developed for the mould design and process simulation studies of the compression moulded SMC;
- (iii) the code of practice-based design of FRP chemical equipment design is being formulated as an "Expert System" programme. The provisions of the British Standards code of practices BS4994 is coded as "IF-THEN" rules and chemical equipment design is being taken up as an Expert System for the formulation of the decision-making process;
- (iv) the sol-gel process is a recently developed method for making high-strength ceramics, which have unique properties including superconductivity. Their production, involving the mixing of organic compounds with metals, could not be done satisfactorily before. In the sol-gel process, a metal can be 'hidden' in an organic compound and the mixture then baked at low temperature such that the organic compound evaporates resulting in a controlled mixture of atoms that high-temperature processing can not produce. A study has been initiated to develop silicon carbide reinforced ceramic systems using this process;
- (v) development of RRIM of casting-grade nylon;
- (vi) the thermoforming of carbon-fibre-reinforced thermoplastic prepregs;
- (vii) development of glass filled nylon cycle parts, and

(viii) for high temperature applications involving thermal environment exceeding 150 °C, the development of matrix and adhesives, material characterization as well as process/fabrication technologies being important, a critical study of high temperature polymer matrix composites is being carried out.

12. Concluding remarks

In this paper, the various R & D activities of the FRP Research Centre (FRPRC) in the last seven years have been highlighted. These research studies cover a wide range of problems. It is worth pointing out that apart from theoretical/analytical research work, the FRPRC has been focussing equal attention on processing/fabrication methods and product development which in the field of composites are very important. This is because of the fact that in the case of composites, the material and the product being developed are totally interlinked, which is not the case when designing with conventional metallic materials. A designer cannot, therefore, design a product without due consideration being given to processing methods and the material system design. Thus product development involving composites will continue to have high priority in future R & D programmes of the FRPRC. Problems of FRP product development for cryogenic applications, high temperature applications, involving thermal environments of 200°C and above, the design and development of an all-composite wing model for a combat aircraft like Rafale, and similar problems will prove challenging and are therefore worth undertaking. It is only when one is confronted with the various challenging problems of designing and developing hardware which has to meet the specifications stipulated by a client or user that all the theoretical/analytical studies are put to practical use and become meaningful. Otherwise, it is something like 'love's labours lost'!

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Analysis of free edge effects in composite laminates

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Abstract. Extensive work has been done in the field of free edge effects in composite laminates. The present paper reviews the modelling as well as the experimental work on this class of boundary value problems. The modelling techniques developed by a number of investigators and the experimental validation of the predictions made through these models are discussed.

Keywords. Free edge effects; composite laminates; boundary value problems.

1. Introduction

The study of delamination phenomena in structural composite laminates began with analytical and experimental observations of the response of such bodies in the vicinity of a free edge. In 1967, Hayashi (1967, pp. 43–48) presented the first analytical model treating interlaminar stresses in what has come to be known as the “free edge problem”. Characteristically, this work focused on the computation of interlaminar shear stress, as in the early stages of composite research interlaminar and delamination effects were viewed as being synonymous with interlaminar shear. The presence of interlaminar normal stress, being of a more subtle origin and also seemingly defying common intuition, was not appreciated until many years after the pioneering work of Hayashi.

The development of the Hayashi model was based upon the implicit assumption that the in-plane stresses within a given layer did not depend upon the thickness coordinate. The magnitude of the maximum interlaminar shear stress was calculated to be a relatively large value in a glass-epoxy [0/90] laminate. Unfortunately, however, owing to the omission of the interlaminar normal stress, the computed stress field within each layer does not satisfy moment equilibrium.

The first reported experimental observations involving free edge delamination were made by Foye & Baker (1970). In that work, tremendous differences in fatigue life of boron-epoxy composite laminates as a function of layer stacking sequence were reported. Severe delaminations were witnessed in that work and were identified as the primary source of strength degradation in fatigue.

From this early work to the present time, the free edge laminate problem has been the most prominent device utilized in the study of composite delamination. Hence, in this paper we shall examine the various models that have been developed through the years to predict the stress field in such a body. Our emphasis shall be placed on the work which led to the development of the global-local model, a model which attempts to circumvent the overwhelming difficulties and complexities associated with stress analysis of multi-layered composite laminates. In this model, 3D elasticity problems are transformed into 2D problems in a self-consistent approach that features realistic satisfaction of boundary and interface conditions. Other methods of analysis, including finite element modelling, are not described in this paper.

The development and use of the global-local model has helped advance the understanding, both quantitatively and qualitatively, of delamination, the most feared mode of failure, in composite laminates.

2. Effective modulus theory

At this point we shall provide a discussion regarding the level of abstraction of the theoretical models treated in this paper. This level of abstraction of modelling detail is called Effective Modulus Theory, or “ply elasticity”, an accurate descriptive term coined by Wang (1987). At the heart of this concept is the representation of each layer within a laminate as a homogeneous, anisotropic, usually elastic body. The laminate itself is viewed as a collection of such layers which are, in most cases, bonded together at their interfaces.

Thus, the ensuing models lead to a piecewise constant representation of the stiffness matrix C_{ij} in the (z) thickness direction, i.e., discontinuities in C_{ij} occur at various interfaces. This form of representation is admittedly artificial; however, it is widely used, almost exclusively so, in practice and research, and has proven to be a valuable tool in composite mechanics.

In a physical composite laminate, each layer is reinforced by hundreds or thousands of fibres per square inch of cross sectional area. Obviously, modelling such details in a precise manner is an impossible task. It is equally clear that the effective modulus predictions cannot be interpreted as providing a precise point-by-point description of the stress field. Only very few studies (Pagano & Rybicki 1974; Rybicki & Pagano 1976) have attempted to study the relationship between “microstresses” (constituent material level stresses) and the stresses given by effective modulus theory in the presence of steep stress gradients. These studies were made approximately a decade ago and were susceptible to errors caused by computer limitations. They were also necessarily of narrow scope; however, they do tend to support the use of effective modulus theory provided the results are averaged over distances comparable to unit cell dimensions. Also note that no geometric singularities were treated in these studies – only the (artificial) free edge singularities of effective modulus theory were considered. Much could be learned by repeating or extending these studies with the aid of modern computer facilities.

There is considerable evidence to suggest the utility, if not precision, of effective modulus theory, even in the presence of macroscopic stress gradients. The solution of the free edge problem and the successful predictions of problems involving local damage such as layer flaws testify to this success. It should also be observed, however,

that the effective modulus models have been correlated with failure by such empirical failure laws as those of classical elastic fracture mechanics and the average stress criterion. This does not prove that these models are fundamentally sound as limited direct experimental evidence regarding the detailed localized influence of these geometric features simply does not exist. Thus the physics of the failure process in composites remains as much a mystery as in homogeneous materials and our understanding of failure is strongly dependent on experimental information. Hence, the use of effective modulus modelling should be viewed as an approximation rather than a fundamental truth and, as such, requires experimental confirmation of the phenomenon being studied.

3. Free edge boundary value problem

Consider the class of boundary value problems in which a prismatic symmetric laminate having traction-free edges at $y = \pm b$ and surfaces $z = \pm h$ is loaded by tractions applied only on its ends $x = \text{constant}$ such that all stress components are functions of y and z alone. Each layer is composed of unidirectional fibre-reinforced material such that the fibre direction is defined by its angle θ with the x -axis where $\theta(z) = \theta(-z)$. We treat each layer as a homogeneous, anisotropic body represented by its effective moduli.

The elastic properties of various composite materials considered in the examples in this paper are shown in table 1. These are not necessarily exact properties measured in laboratory tests, but are values that have been used in conducting analytical studies throughout the course of the work reported herein.

Table 1. Composite materials used.

Property	Material I	Material II
	Graphite/ epoxy	Graphite/ epoxy
E_L (MSI)*	20.0	25.1
E_T (MSI)	2.1	4.8
E_z (MSI)	2.1	0.75
G_{Lz} (MSI)	0.85	1.36
G_{Lz} (MSI)	0.85	1.2
G_{Tz} (MSI)	0.85	0.47
ν_{LT}	0.21	0.04
ν_{Lz}	0.21	0.25
ν_{Tz}	0.21	0.17

Here E and G symbolize Young's modulus and engineering shear modulus, respectively. The subscripts denote the L , T , z directions, where L is the fibre direction, T the in-plane transverse direction, and z the thickness direction, ν_{ij} is the Poisson ratio measuring strain in the j direction caused by uniaxial stress σ_i .

* 10^6 psi = 6.895×10^9 Pa.

4. Analytical methods

A number of techniques, with varying degrees of sophistication, have been used for the solution of the boundary value problem. The following methods, developed by a team of professionals active in this area of specialization, have been used for studying this problem:

- (i) finite difference method (Pipes & Pagano 1970),
- (ii) primitive delamination model (Pagano & Pipes 1973);
- (iii) elastic plate model for σ_z (Pagano 1974);
- (iv) Reissner's extended variational principle (Pagano 1978); and
- (v) global-local laminate variational model (Pagano & Soni 1983).

A brief description of these methods is given in the following sections; for details the reader is referred to Pagano & Soni (1983) which contains detailed treatment of these models.

4.1 Finite difference solution

The first attempt to solve the free edge problem by use of the theory of anisotropic elasticity without any simplifying assumptions was given by Pipes & Pagano (1970). In that presentation, a laminate consisting of four unidirectional fibrous composite layers, two with their axes of elastic symmetry (fibre direction) at $+\theta$ and two at $-\theta$ to the longitudinal laminate axis, was considered. Figure 1 shows the laminate geometry and coordinate system. Each layer is of thickness h_0 . The constitutive relations for each layer with respect to the laminate coordinate axes are used in which the thirteen anisotropic material constants are related to the nine constants with respect to the material symmetry axes through the well-known stiffness transformation law. The use of layer strain-displacement relations in equilibrium equations provide three coupled partial differential equations.

The field equations form a set of elliptic, coupled, second-order partial-differential equations. The governing equilibrium equations are solved in conjunction with relevant boundary conditions using the finite difference method. This method of solution has been well established as a technique for obtaining numerical solutions for elliptic partial differential equations (Forsythe & Wasow 1960). When the finite difference technique is employed, the continuous material region is replaced by a system of discrete material points where discrete values of the dependent variables are to be determined. The partial differential equations are expressed at each material point within the region as algebraic equations by approximating partial derivatives by finite-difference operators. In this work, central difference operators were employed at interior region material points, while forward and backward difference operators were employed at material points falling on boundaries. When the partial differential equations and boundary conditions are written in their approximate form as finite-difference equations, a linear, nonhomogeneous system of algebraic equations results. The number of algebraic equations is equal to the product of the number of material points in the material region and the number of dependent variables. By storing only those terms of the coefficient matrix which fall within the band width of non-zero coefficients in the digital computer, a simple Gauss elimination scheme can be employed to solve large numbers of algebraic equations with maximum economy.

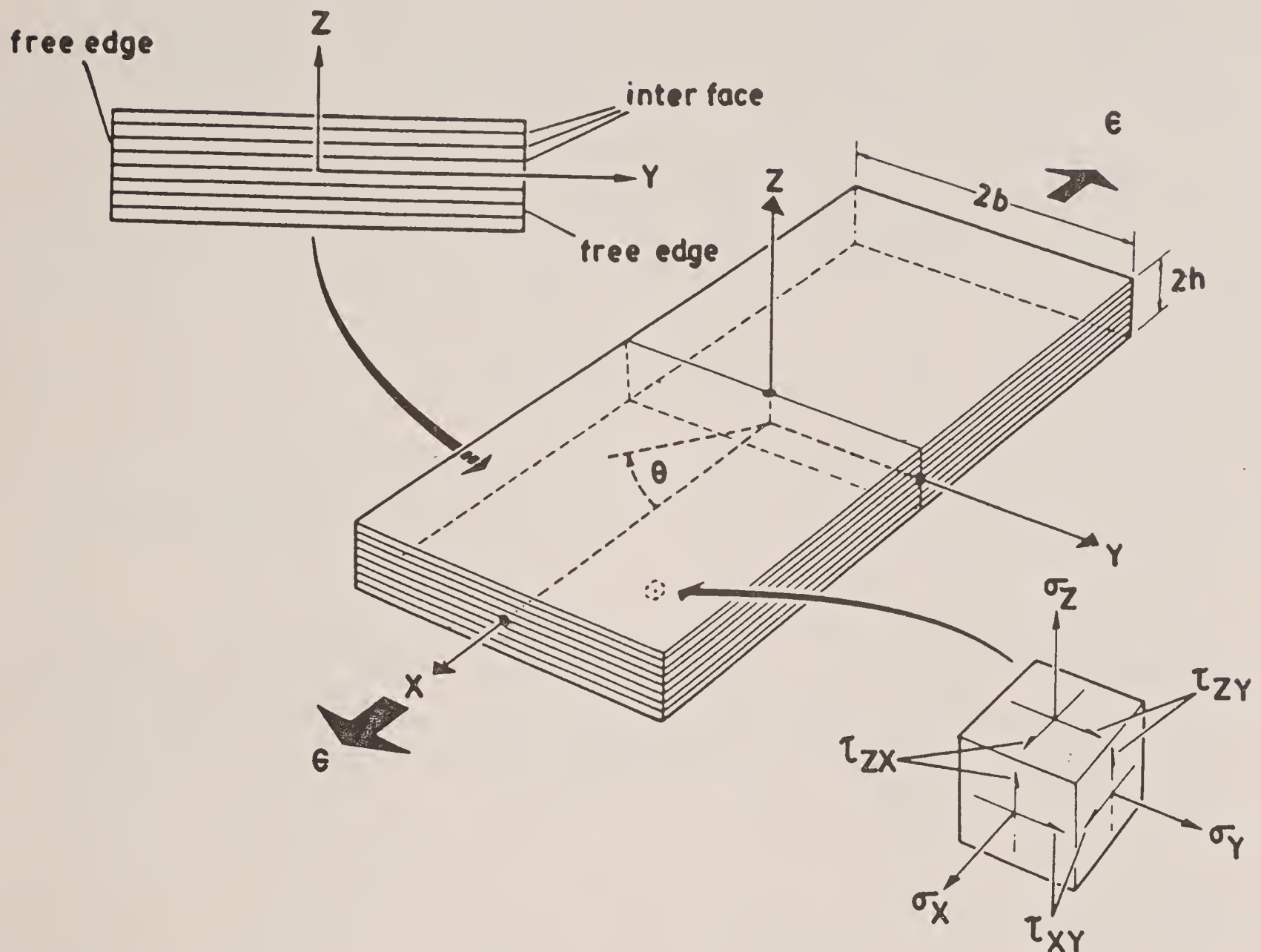


Figure 1. Laminate geometry.

The material region is reduced by symmetry conditions to that of one quadrant of the laminate cross-sectional region in the yz -plane. The continuous region is replaced by a rectangular grid of material points with constant, square grid spacing. The interface continuity conditions at $z = h_0$, which require continuity of the functions $u, v, w, \sigma_z, \tau_{xz}$, and τ_{yz} , are approximated by constraining the interface to lie midway between two rows of material points.

Numerical results for τ_{xz} are presented in figure 2 and show the functional dependence of the interlaminar shear stress on fibre orientation. The ordinates of this curve are the respective normalized values of τ_{xz} at the node point on the free edge nearest the interface. It is interesting to note that a sign reversal in this quantity occurs near $\theta = 60^\circ$. This figure also shows the results computed by model (iv) entitled "Reissner's extended variational principle" (Pagano 1978) with each layer modelled separately. The comparison between the results by the two methods is very good. Further refinement of results can be made by increasing the number of subdivisions of each layer in the computation by both methods of solution.

4.2 Primitive delamination model

In order to utilize these previous results to determine the delamination resistance of a specific laminate, it is necessary to provide a model to compute the interlaminar stresses in the body. A convenient, albeit crude, model was developed (Pagano & Pipes 1973)

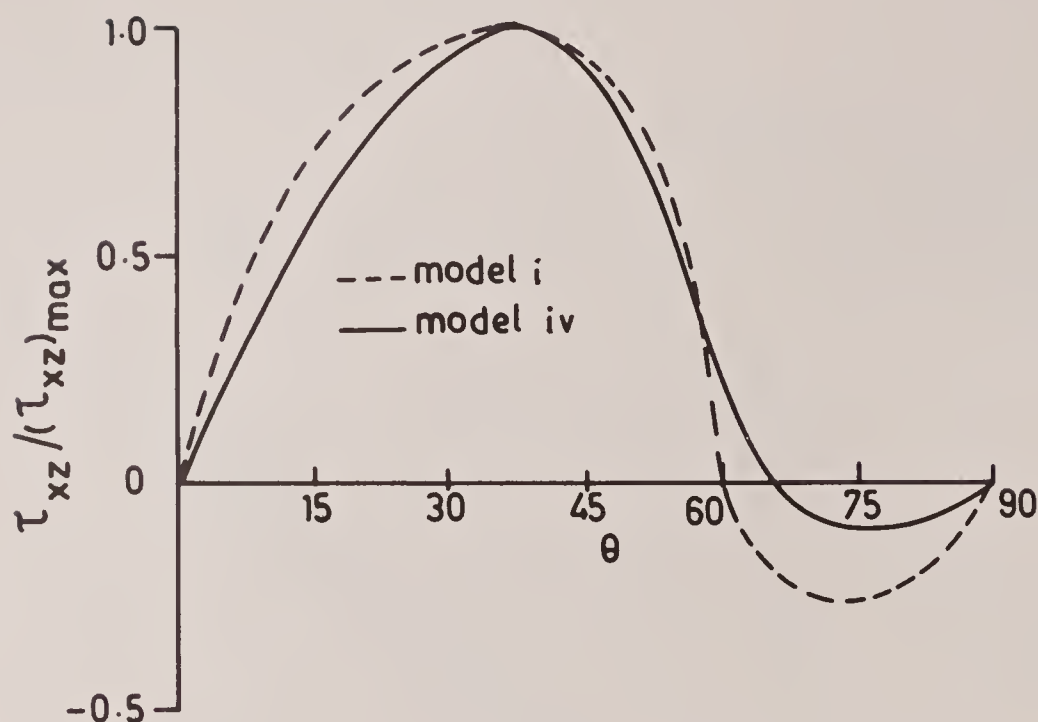


Figure 2. Comparison of τ_{xz} in $(\pm\theta)_s$ laminates at $-\theta/\theta$ interface.

based on the approximation for σ_z shown in figure 3, where the maximum value, σ_m , occurs at the free edge. The model also assumes that σ_z differs from zero only in the boundary layer region, i.e., within a distance equal to the laminate thickness $2h$ from the free edge. Since the resultant of this distribution must be a pure couple, the following relation must be satisfied

$$\sigma'_m = \sigma_m/5, \quad (1)$$

while σ_m is given by

$$\sigma_m = \sigma_m(z) = 45 M(z)/14h^2, \quad (2)$$

where

$$M(z) = \int_z^h \sigma_y(\xi)(\xi - z) d\xi \quad (3)$$

and $\sigma_y(z)$ is the transverse stress given by lamination theory. Note that (3) is the corrected version of a relation that was erroneously stated in Pagano & Pipes (1973). Estimates of interlaminar shear stresses to this level of approximation can also be derived, but there has been no urgency associated with them. Hence these relations are not shown here.

The above discussion has outlined an approach to predict how layers of specific

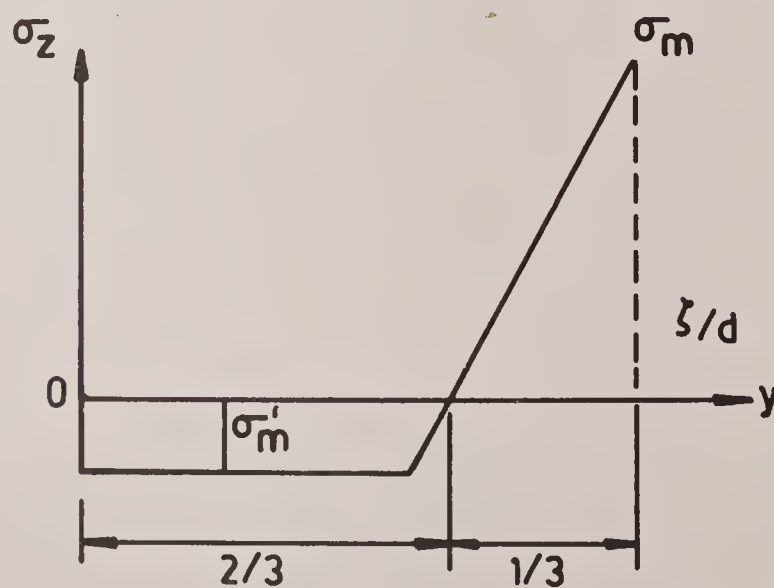


Figure 3. Assumed distribution of σ_z across laminate width.

orientations should be arranged through the thickness to provide optimum protection against delamination in laminated coupons. We should note that this work, in conjunction with the experiments of Foye & Baker (1970), demonstrates that, depending on the specific laminate being characterized, the use of flat laminated coupons may be inadmissible for fatigue testing, and possibly monotonic tension or compression as well. This observation stems from the fact that the desired failure mode may not take place due to free edge effects. In fact, free edge effects may dominate the entire failure history of the laminate.

4.3 Elastic plate model for σ_z

In this section we shall review the development of a model by Pagano (1974) to calculate the interlaminar normal stress distribution along the central plane of the free edge laminate. The concepts and results shown here have proven invaluable in leading to the establishment of the global-local model for laminate response. The model consists of the treatment of the quarter-section of the laminate as a plate fastened to a rigid foundation. As before, the body is subjected to a constant axial strain ϵ and a constant temperature rise T .

Owing to the symmetric lamination geometry, the vertical displacement w and the interlaminar shear stresses τ_{xz} and τ_{yz} all vanish along the central plane $z = 0$. Guided by this observation and taking account of the usual aspect ratios of a composite test specimen, i.e., $b/h \gg 2$, we attempt to define the response by treating the upper half of the laminate, $z \geq 0$, as a plate under cylindrical bending, where all force and moment resultants are independent of x . In this model, the surface traction $q(y) = -\sigma_z(y, -h/2)$ acting on the mid-plane is treated as a dependent variable.

We may now reflect on the nature of the plate theory required to provide an accurate calculation of $q(y)$. For example, if we solve the boundary value problem illustrated in figure 1 by the use of classical lamination theory, we find that q vanishes identically. Therefore, we must consider a higher order theory, such as the shear deformation theory development by Yang *et al* (1966) and modified by Whitney & Pagano (1970). In this formulation, however, the displacement w is independent of z , hence the prescription of w at the base of the plate fixes w everywhere and, in particular, at the ends $y = 0, b$. In this case, we are not free to prescribe the vertical shearing force resultant at both ends so that the resultant of the distribution of $q(y)$ differs from that given by exact analysis. This approach then must also be rejected. We conclude that the simplest appropriate theory must include shear deflection plus thickness stretch deformation. Such a theory has recently been presented by Whitney & Sun (1973); a slightly modified version of that theory has been employed in this work. The solution to the governing equilibrium equations for the class of boundary value problem stated in §2 is derived in closed form. The distribution of stress component σ_z for a $(0/90)_s$ laminate calculated on the basis of model (i) and this model (iii) is given in figure 4. This model is unable to make accurate predictions of the other interlaminar stress components and, hence, necessitated the development of the model on the basis of Reissner's variational theorem (Reissner 1950).

4.4 Reissner's extended variational principle

The approach suggested by Pagano (1974), i.e., treatment of each layer as a plate

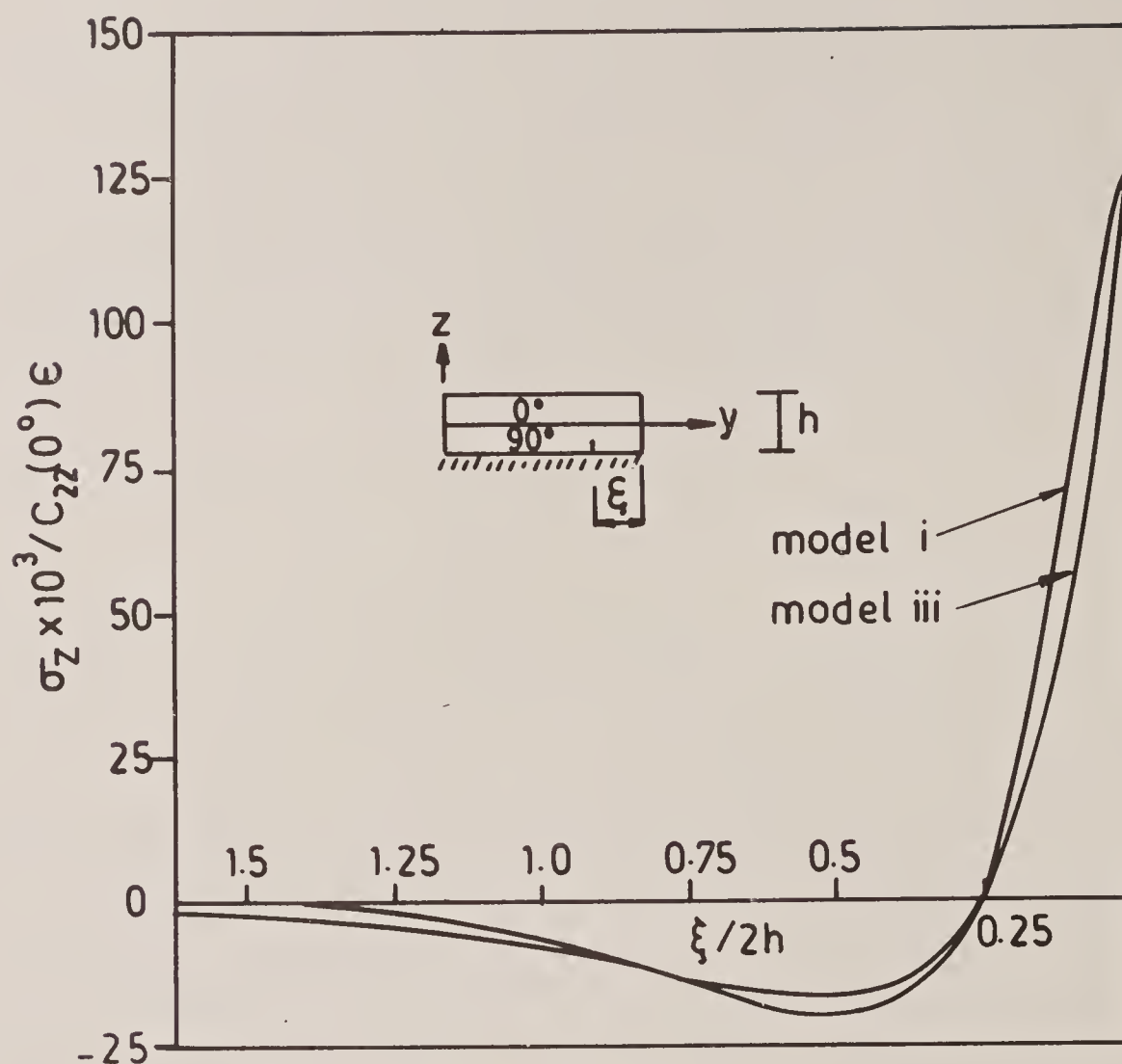


Figure 4. Distribution of σ_z along central plane.

governed by the Whitney-Sun theory (Whitney & Sun 1973), lacks generality since it can be shown that less than 5N edge traction boundary conditions are available in that approach. Furthermore, as a consequence of interface displacement continuity, the natural edge traction boundary conditions are coupled, i.e., they involve functions of the tractions acting on two (adjacent) layers. Also, the layer interlaminar tractions are independent of the constitutive equations. Thus, extension of this approach by allowing higher-order displacements is not advisable, since, as in the previous approaches, proper equilibrium of each layer under its prescribed tractions cannot be enforced.

The previous discussion defined a clear need to examine new approaches for laminate stress analysis since all known approximate laminate theories at the time (1978) were based upon assumed displacement fields, which as we have seen, lead to results lacking credibility. Therefore, requirements to be satisfied by an acceptable laminate field theory were set down and a self-consistent model (Pagano 1978) developed in accord with the requirements, which are: a) all six stress components are non-zero in general; b) traction and displacement continuity conditions at interfaces between adjacent layers are satisfied; c) the principle of "layer equilibrium" must be satisfied. This principle is defined as follows. Consider a region within the laminate that is arbitrarily located except that it is bounded by any two of the parallel interfaces of the laminate. It is required that the computed stress field acting on the surfaces of the arbitrary region, in conjunction with the prescribed traction boundary conditions (pointwise, in the elasticity sense) on those portions of the external laminate boundary which lie in the region, satisfy the conditions of vanishing resultant force and moment identically. Thus, every layer must satisfy this requirement, which is referred to as the principle of layer equilibrium. This implies that the appropriate force variables in the field theory are force and moment resultants (per unit length) acting on the cross sections of a layer

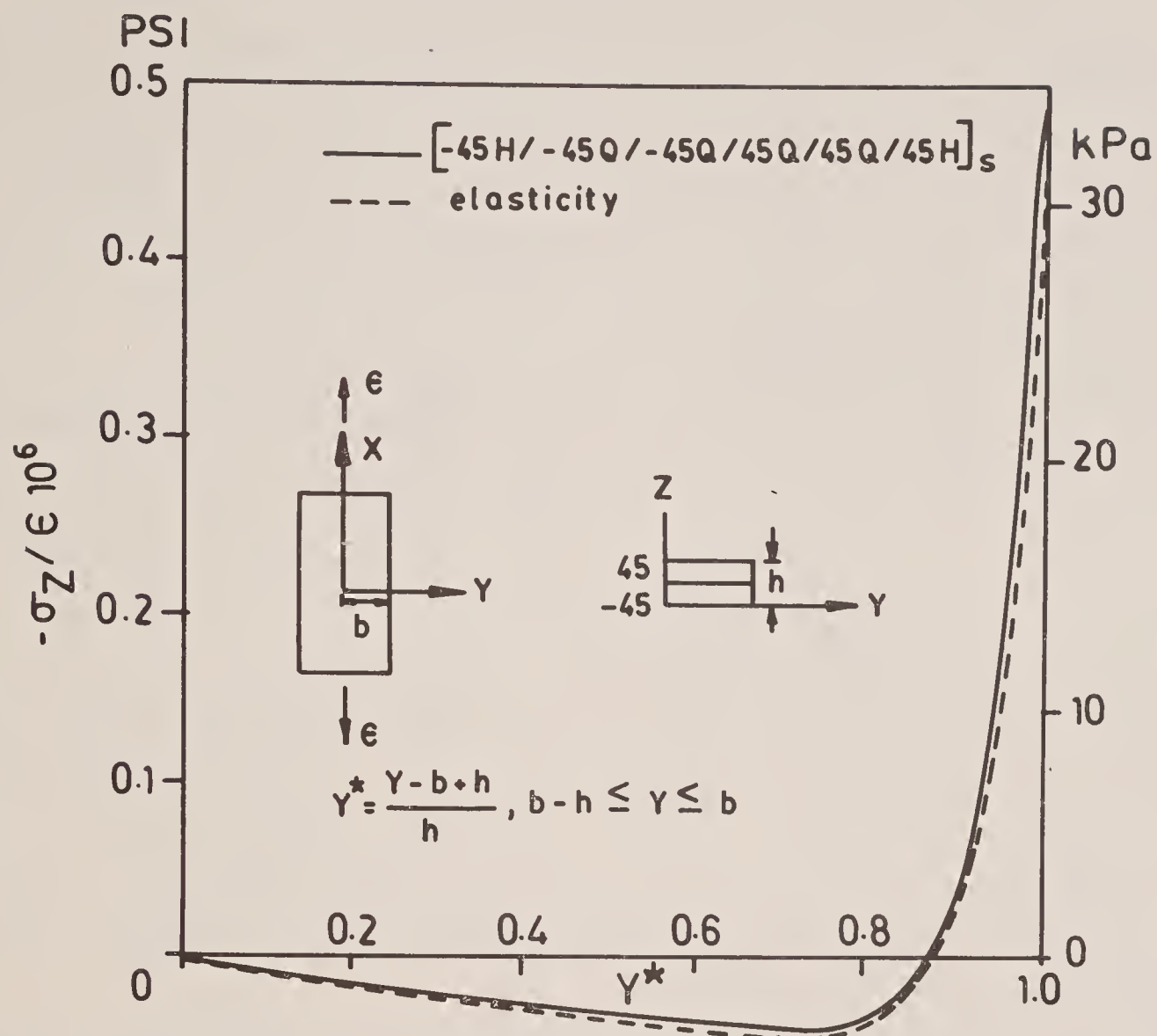


Figure 5. Comparison of stress component σ_z at the ± 45 interface between results from the elasticity model (Wang & Choi 1982) and the present model, material 1.

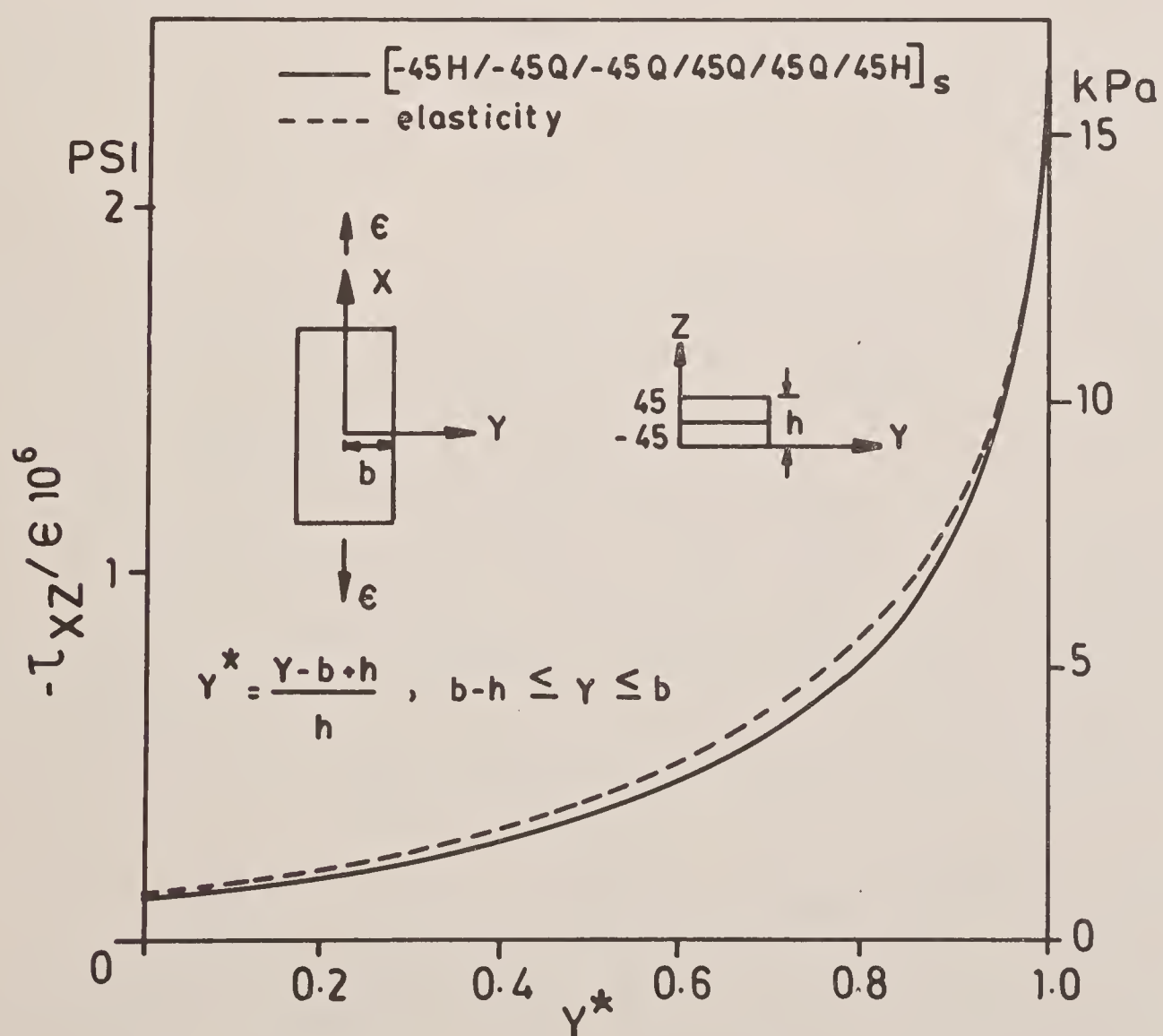


Figure 6. Comparison of stress component τ_{xz} at the ± 45 interface between results from the elasticity model (Wang & Choi 1982) and the present model, material 1.

and interlaminar stresses on its interfacial surfaces. Although sub-regions not bounded by interfacial planes need not satisfy layer equilibrium, in problem solving, additional interfaces may be introduced conceptually to improve solution accuracy.

Although the above requirements do not define a unique formulation, the simplest model within this class was derived. The model permits the treatment of discontinuous interfaces, i.e., interface cracks. It also forms the basis for the global-local model, which we shall treat in a subsequent section.

To develop this model, a laminate which is built of anisotropic elastic layers of uniform thickness and is subjected to prescribed tractions and/or displacements on its boundary is considered. The body is bounded by a cylindrical edge surface and upper and lower faces that are parallel to the interfacial planes. Since it is necessary to consider both traction and displacement continuity conditions at the various interfaces, it is logical to examine Reissner's variational theorem (Reissner 1950) as a mechanism to develop the appropriate field equations. Reissner has shown that the governing equations of elasticity can be obtained as a consequence of the variational equation. In Pagano (1978) the stress components are assumed to be functions of z and moment and stress resultants. The substitution of these parameters in the variational functional leads to the required set of equilibrium equations, constitutive relations, interfacial continuity conditions and edge conditions. These equations are solved for a number of laminates to predict the stress distribution in each layer and interface due to uniaxial in-plane loading conditions.

We now turn to the elasticity solution for the $(\pm 45)_s$ laminate given by Wang & Choi (1982) using material 1 properties. In that work, an exact expression for the stress singularities is derived while the field solution itself is determined numerically. In figures 5 to 8, attention is focused on the interface between the $\pm 45^\circ$ plies as we compare the two solutions for the distribution of stresses across the width of the laminate. Results from the present model are based on the stacking sequence $(-45H/-45Q/-45Q/45Q/45Q/45H)_s$, where the symbols Q and H stand for quarter- and half-layer thickness, respectively. The two models agree very well for the calculations of interlaminar stresses σ_z and τ_{xz} as shown in figures 5 and 6. The computed distributions of in-plane stresses σ_x and τ_{xy} , however, demonstrate some significant disagreement. What is not shown in figures 7 and 8 is the fact that the elasticity solution for each of these functions is singular at the free edge. However, the singular zone is so small that it cannot be shown on the given scale. Observe that the present model prediction for τ_{xy} satisfies the traction-free boundary condition while the elasticity result may not.

The occurrence of very large numerical values of the intermediate parameters during computation of results in the case of a large number of layers causes computer overflow problems. This in turn restricts the application of the model for investigating thick laminates. For example, the maximum number of layers in the analysis of laminates treated in the technologies of the 1970's was 12. A larger number of layers may be considered by the use of a global-local model discussed later.

4.5 Global-local laminate variational model

The absence of a unified, tractable model to predict the elastic response of a multi-layered laminate (say 100 layers) has foiled attempts to understand the failure modes of practical composite structures. Global models (i.e., model iii), which follow from an assumed displacement field and lead to the definition of effective (or smeared) laminate

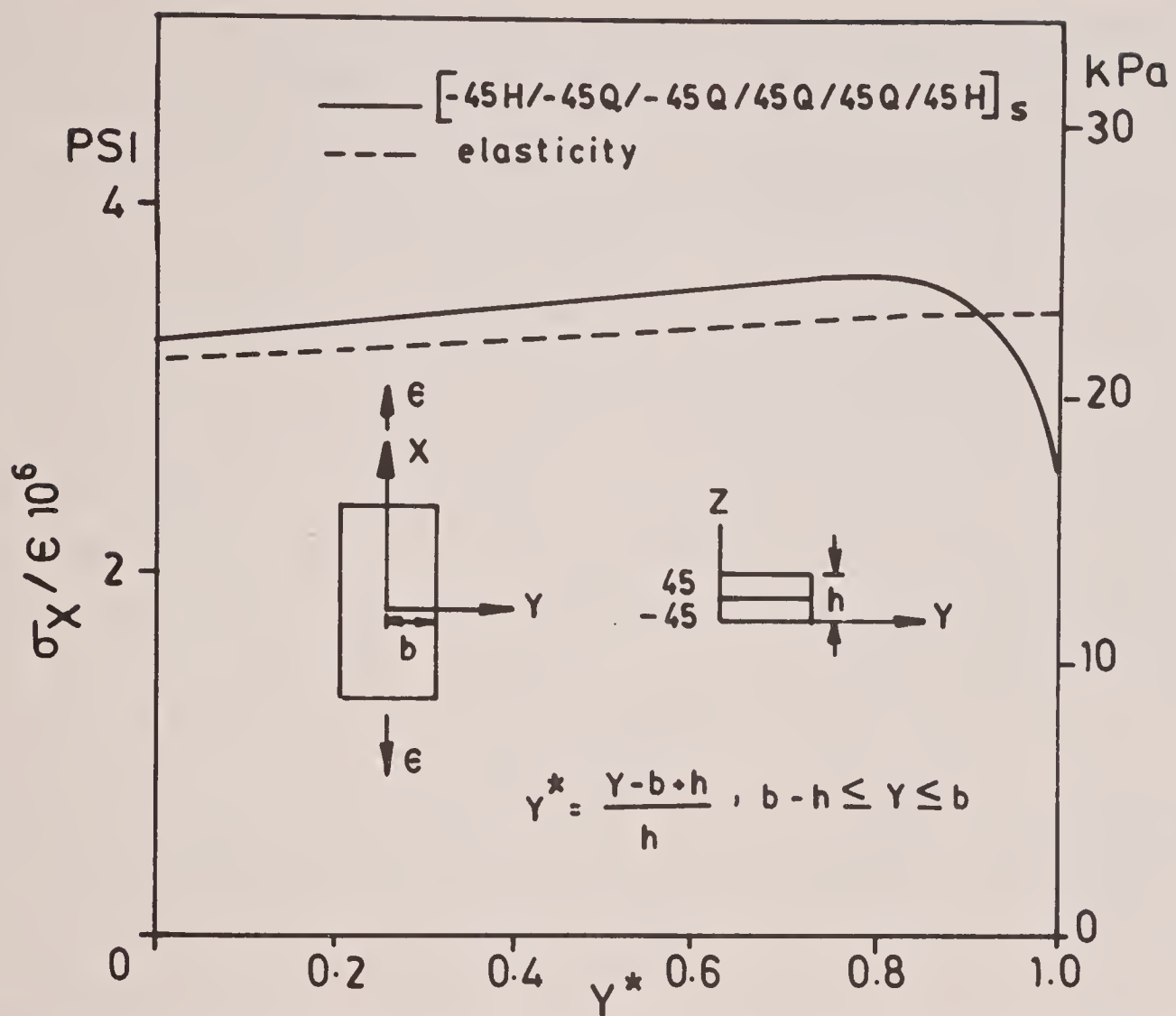


Figure 7. Comparison of stress component σ_x at the ± 45 interface between results from the elasticity model (Wang & Choi 1982) and the present model, material 1.

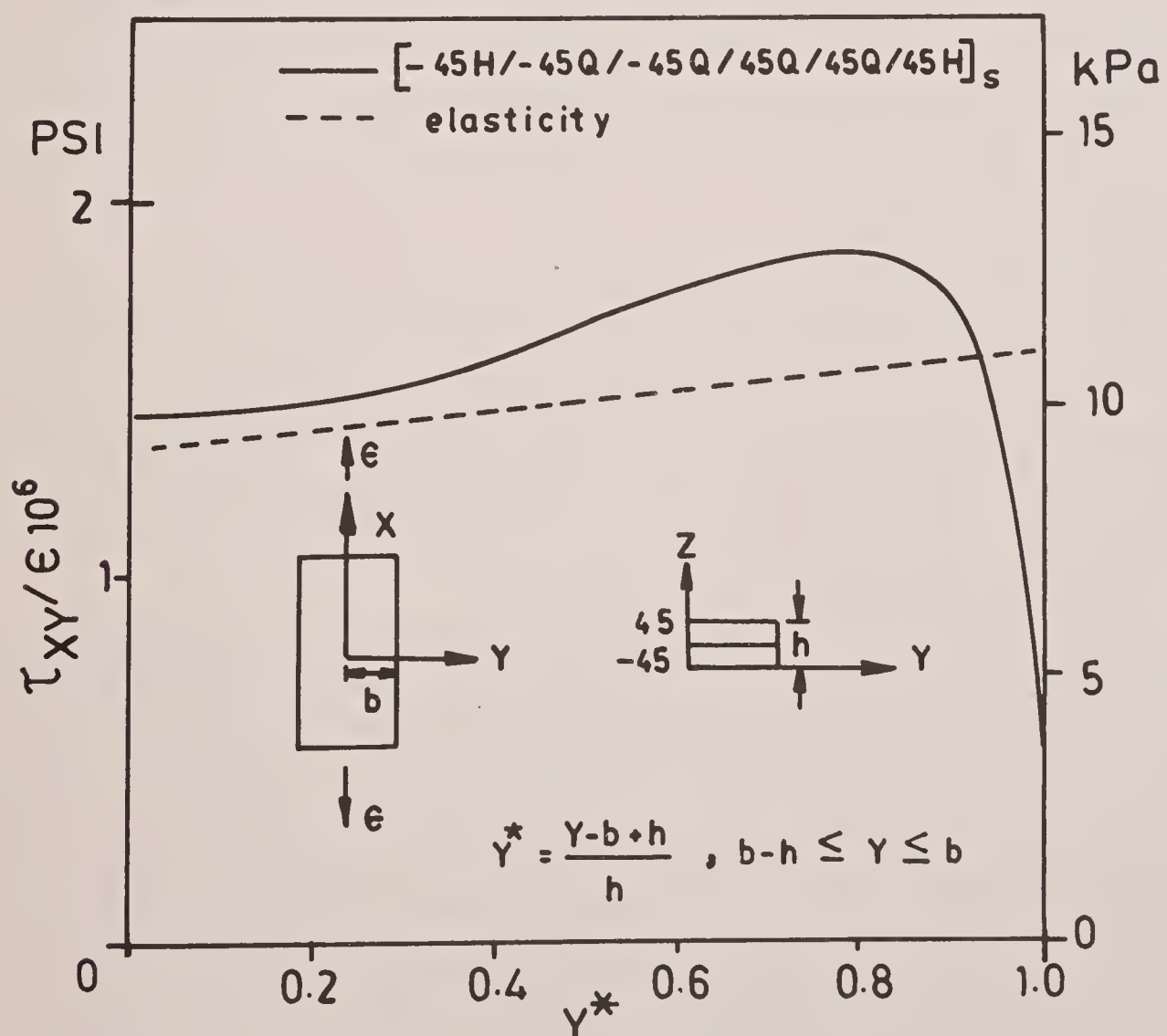


Figure 8. Comparison of stress component τ_{xy} at the ± 45 interface between results from the elasticity model (Wang & Choi 1982) and the present model, material 1.

moduli, are not sufficiently accurate for stress field computation. On the other hand, local models (i.e., models i and iv), in which each layer is represented as a homogeneous anisotropic continuum, become intractable as the number of layers becomes even moderately large, as we have just seen. Pagano & Soni (1983) blended these concepts into a self-consistent model which can define detailed response functions in a region of interest (local), while representing the remainder of the domain by effective properties (global). In this investigation the laminate thickness is divided into two parts. A variational principle has been used to derive the governing equations of equilibrium. For the global region of the laminate, potential energy has been utilized, while the Reissner functional has been used for the local region. The field equations are based upon an assumed thickness distribution of stress components within each layer of the local region and displacement components in the global region. The derived boundary conditions imply that the computed stress field on the surfaces of the global region and the prescribed tractions (pointwise in an elasticity sense) satisfy the conditions of vanishing resultant force and moment identically. The same conditions are satisfied in the local region. The self-consistent global-local model has been developed to investigate the stress fields in laminated media consisting of numerous layers. This model defines detailed response functions, such as interlaminar stresses and single layer forces and moments in a pre-determined region of interest (local), while the remainder of the domain is represented by its effective material properties and the corresponding resultant forces and moments (global). The local model employs a theory (Pagano 1978) which approaches the theory of elasticity in the limit of vanishing layer thickness. The global model is based upon an approach by Whitney & Sun (1973) which has been demonstrated to produce good agreement with elasticity results on the global boundary for a particular laminate by Pagano (1974). While a particular arrangement of global and local domains had been considered in the computation of results, there is no difficulty in extending the program to include more general arrangements, including the use of more than one global domain. The importance of the latter option follows from the observation that model accuracy may be improved by a gradual rather than abrupt transition of region.

The effectiveness of the model has been demonstrated by the use of numerical examples based upon the free-edge class of boundary value problems in laminate elasticity. Preliminary results have been very promising although an apparent loss in accuracy occurs in the calculation of stress components of small magnitude, which may thus require finer subdivision of the local region than would normally be required. Similarly, the effect of the aforementioned "transition region" on model precision will require further study.

It is clear that theories of the type presented here are needed to describe the response of laminated structural components used in practice. More results defined by the global-local model and its use in the definition of interlaminar failures are given by Soni and Kim (Kim 1982; Soni 1982; Kim & Soni 1984, 1985; Soni & Kim 1984, 1987). In these references, the influence of interlaminar shear and tension on delamination are treated. Experimental investigations are carried out to determine interlaminar normal stress at the mid-plane of laminates, delamination stimulated by stress component σ_z and/or τ_{xz} and procedures to suppress free edge delamination. During these investigations a large number of previously unknown aspects of layered composite materials have been discovered (such as: vanishing effect as stress rises at free edge for laminates with low width-to-thickness ratio, averaged stress components near

the free edge region over a distance along the width equal to a ply thickness as effective stress components and the free edge delamination stimulated by interlaminar shear stress). The model has been quite useful in studies of material, geometric, and lamination parameters and their influence on the interlaminar response of laminates containing free edges. A new solution algorithm is presently being developed to treat more general problems in laminate elasticity.

An interesting observation has been made regarding the class of laminates considered here, i.e., those possessing very small values of width-to-thickness ratio. In such cases, the width is insufficient to promote the appropriate stress transfer mechanism necessary to develop the stress distribution given by classical lamination theory. Aside from the stress prediction, even the effective laminate properties may differ from those given by classical lamination theory. These points have been demonstrated by the examination of a similar class of boundary value problems well-known in composite mechanics literature, i.e., the free edge problem in laminate elasticity as the width-to-thickness ratio approaches zero, as shown in figure 9. To examine this limiting case, consider the response of two laminates, $(0/90)_s$ and $(\pm 30)_s$, as a function of aspect ratio according to the model of Pagano (1978). Each laminate is subjected to a constant longitudinal strain $\epsilon_x = \epsilon$. In the former laminate, the transverse stresses σ_y , σ_z and τ_{yz} dominate the response, while in the latter, τ_{xy} and τ_{xz}

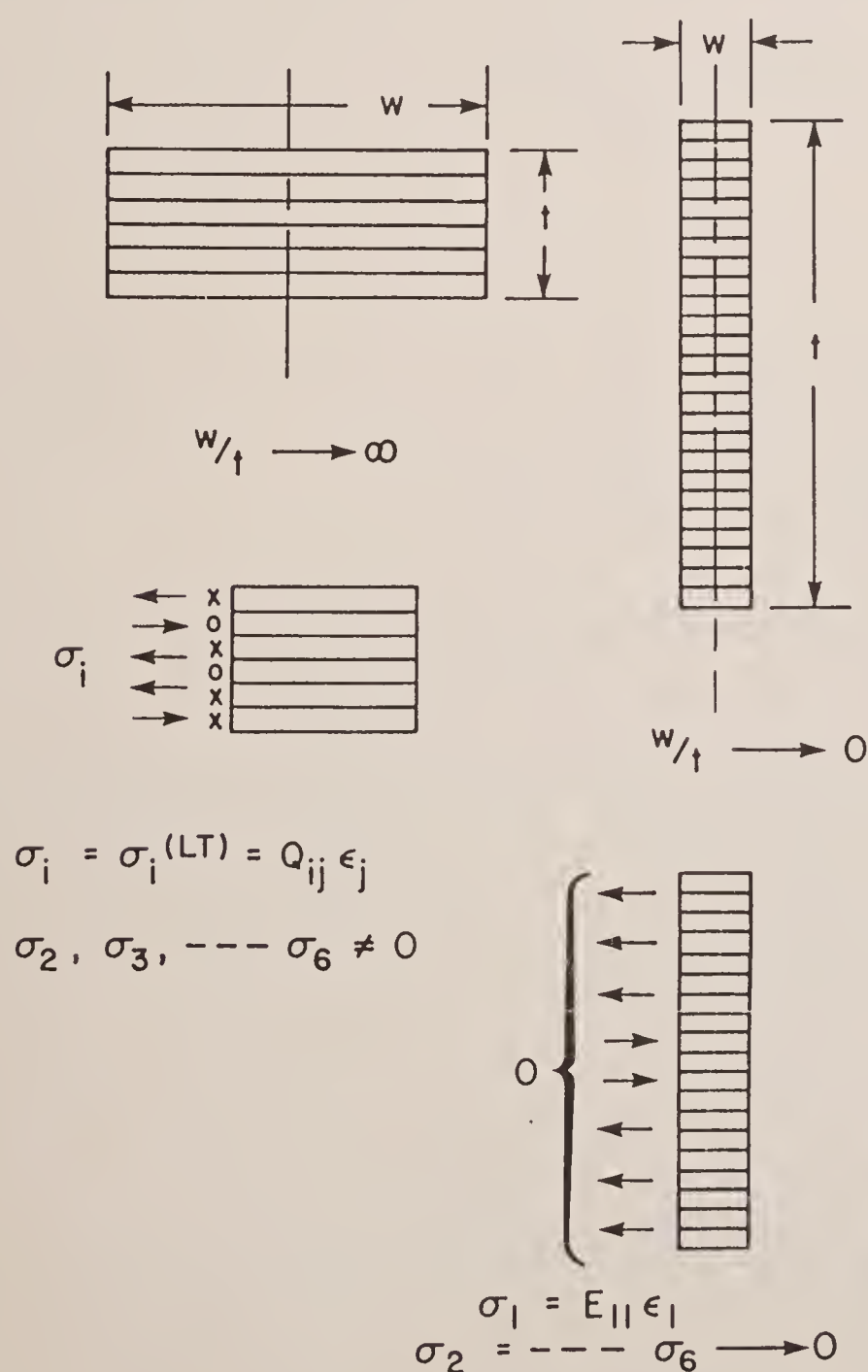


Figure 9. Width/thickness effects in axial loading.

Table 2. Maximum normalized stresses σ^* and laminate modulus, $(\pm 30)_s$ -laminate, material II (see figure 1).

w/t	σ_y^*	σ_z^*	τ_{xy}^*	τ_{yz}^*	τ_{xz}^*	E_x^*
4.0	0.01	0.13	5.4	0.03	6.2	11.3
1.0	0.13	0.15	5.2	0.006	5.8	9.0
1.5	0.001	0.13	4.9	0.002	4.9	6.4
0.1	0.002	0.014	1.6	0.001	2.5	6.0

predominate. The properties of material II of table 1 are assumed. The model Pagano (1978) upon which this parametric study is based is an approximate theory that becomes more accurate when each layer is (mathematically) subdivided into sub-layers. The results reported here are based upon 2 subdivisions of each layer. Comparison of the 2- and 3-subdivision cases shows that the reported stresses are reasonable, but not perfect; however, the trend is expected to be correct. Also, this class of boundary value problem contains elastic stress singularities (Wang & Choi 1982) which do not occur in the present model. The rationale and application of such results for practical analysis is discussed in Soni & Pagano (1982). The results of the parametric study are given in tables 2 and 3. Here, the maximum magnitudes of the significant normalized stress components and the effective laminate modulus E_x are presented. The

Table 3. Maximum normalized stresses σ^* and laminate modulus, $(0/90)_s$ -laminate, material II (see figure 1).

w/t	σ_y^*	σ_z^*	τ_{yz}^*	E_x^*
4.0	0.116	0.039	0.04	15.0
1.0	0.17	0.023	0.036	15.0
0.5	0.13	0.011	0.031	15.0
0.1	0.001	0.003	0.005	15.0

normalized stresses are denoted by an asterisk such that

$$\sigma^* = 10^{-6}(\sigma/\varepsilon) \text{ psi}, \quad E_x^* = 10^{-6} E_x \text{ psi},$$

where ε is the (constant) applied axial strain. The results in the tables demonstrate the downward trend of the transverse stresses as the aspect ratio goes to zero. The reported values are the maximum values calculated at 21 locations along the width of the laminate and since the maxima in different laminates may vary with the locations corresponding to each laminate the trend in the tables is slightly variable.

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Theoretical modelling of laminated composite plates

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Abstract. Formulation of appropriate governing equations, simpler than the three-dimensional equations of elasticity yet capable of predicting, fairly accurately, all important response parameters such as stress and strain, is attempted in modelling a structural component. Several theoretical models are available in the literature for the analyses of plates. The emergence of fibre-reinforced plastics as an attractive form of structural construction, added a new complexity to the modelling considerations of laminates by requiring the estimation of the interlaminar stresses and strains. In this paper, modelling considerations of laminated composite plates are discussed. The classical laminated plate theory and higher-order shear deformation models are reviewed to bring out their interlaminar stress predictive capabilities, and some new modelling possibilities are indicated.

Keywords. Laminated composite plates; analyses of plates; fibre-reinforced plastics; interlaminar stress and strain; shear deformation models.

1. Introduction

In general one may define a structure as a material system intended to carry loads, and the structural element as its component. Structural elements are usually categorized for convenience into beams, plates, shells, plane problems etc. depending upon the geometry and loading pattern. The set of equations, whose solution can be interpreted to get response parameters such as stresses and strains, describing the behaviour of the structural element is referred to here as the theoretical model or simply the model. If rigour is the only criterion, the three-dimensional elasticity model is the only valid formulation for all these categories of problems. The complexity of three-dimensional equations, defying the possibility of any reasonable solution for a large class of practical problems, motivated the search for simpler sets of equations, each applicable for a particular structural element and capable of yielding all important response parameters reasonably accurately. Such solutions will naturally not coincide with the three-dimensional solution in all details. The aim is to formulate these simpler sets of equations such that they yield results as close to the solution of the three-dimensional equations of elasticity as possible. Simplicity and accuracy, although contradictory, have been the favourite objectives in shaping theoretical models. Identification and

incorporation of appropriate assumptions, reflecting the physical behaviour of a structural element, into the three-dimensional theory guided the development of theoretical models. The simple assumption of Kirchhoff, that normals to the midsurface of a plate before deformation will remain normal even after deformation, brought about far-reaching simplicity in the model and shaped the plate theory.

In what follows, we will be concerned mainly with modelling of plates. Within the frame work of the three-dimensional theory of elasticity, a plate subjected to transverse loading requires the solution of three simultaneous partial differential equations or its equivalent along with appropriate boundary conditions. On the other hand the classical plate model, based on Kirchhoff's assumption predicts plate behaviour fairly accurately as the solution of the biharmonic equation in normal deflection with appropriate boundary conditions. As a consequence of the simplification, some inconsistencies arise. Some of them are

- (i) transverse shear and normal strains are zero,
- (ii) constitutive law is violated,
- (iii) there is no provision for adequate description of boundary conditions at edges,
- (iv) there is no provision for considering boundary conditions on plate surfaces.

Recognizing the importance of the transverse shear strain in thick plates, shear deformation theories with provision for non-zero transverse shear strain were developed to extend the theory to thick plates (Reissner 1944; Mindlin 1951). The problem of providing adequate description of the boundary conditions at edges is resolved, once the shear strains are accommodated. Unfortunately there appears to be no general way to model plates satisfying plate surface boundary conditions. Krishna Murty (1977), Levinson (1980), Krishna Murty & Vellaichamy (1988a) attempt to model the plates satisfying the zero transverse shear strain conditions at plate surface and Vijaya Kumar & Krishna Murty (1988a) examine the modelling of plates satisfying the normal stress condition at the plate surface also, using the Lagrangian multipliers. These typical developments in modelling isotropic plates provide a basis for modelling laminated plates.

The advent of composites as the most attractive engineering material system, forced a fresh look at the modelling considerations of laminates. In these materials the interlaminar zone is the weakest link as it is essentially a thin layer of homogeneous resin medium. The estimation of interlaminar stresses and strains has become essential in ensuring laminate integrity. Thus a fresh look at the plate theory, becomes necessary to expose the interlaminar stress predictive capabilities of existing models and, if required, to remodel the laminates.

We shall restrict our further discussion to the modelling of fibre-reinforced plastic laminates. Modern fibre-reinforced plastic materials are essentially bundles of fibres embedded in resin. The fibre strength is very high compared to the resin. Diameters of the fibres are of the order of microns. When prepreg material is used to build structural components, which is perhaps the most popular method of building composite laminates currently, we have layers or plies of unidirectional fibres embedded in resin, in a partially polymerized form. A number of such plies are put together to obtain the necessary thickness and are cured under controlled temperature and pressure conditions to build the structural components. Therefore, it is necessary to fix up the type of abstraction for modelling the laminates. Two levels are readily apparent. In order to consider localized details such as the stress field around a fibre, fibre matrix

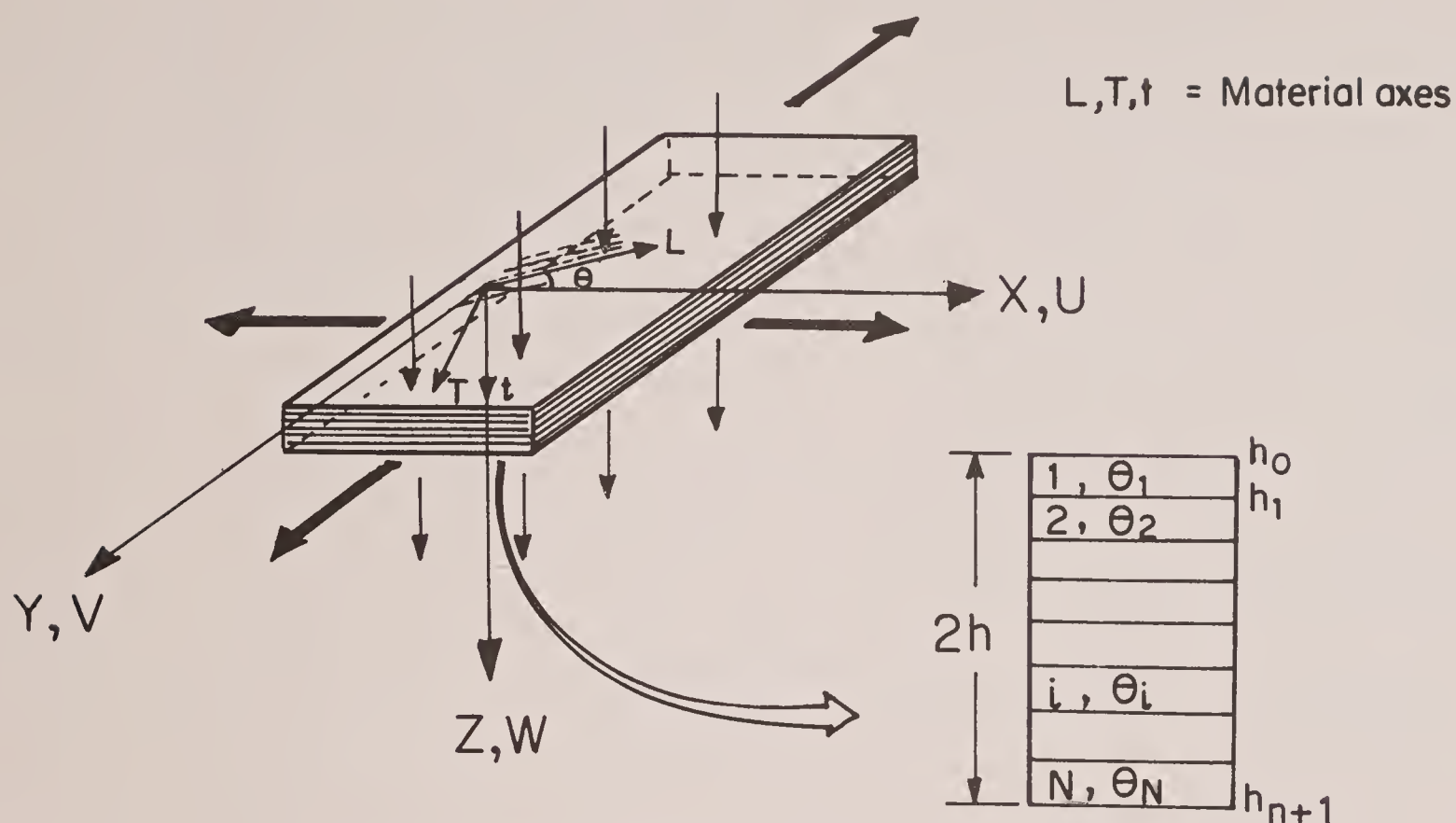


Figure 1. A typical laminate – its geometry and coordinates.

interactions etc., no simplifications are possible and full elasticity equations are the obvious choice. On the other hand, by restricting the attention to certain gross aspects, such as ply and interlaminar strength, one may consider the laminate as a number of layers of an orthotropic homogeneous medium, perfectly bonded at the surfaces such that no slip is possible. Here we consider the second abstraction, and generally, as is well-known, this kind of abstraction provides valuable information of direct use in engineering applications, in assessing the strength, stiffness and vibrational behaviour of laminates.

A typical laminate is shown in figure 1. A look at some of the exact solutions available (Pagano 1970), will reveal the special complexity in modelling laminates. Unlike metals, laminates contain interfaces across which the material constants are discontinuous in the direction of thickness. As a consequence, some of the strain and stress components are discontinuous across the interface, as indicated in table 1.

We shall refer to $(\epsilon_x, \epsilon_y$ and $\epsilon_{xy})$ and $(\sigma_x, \sigma_y, \sigma_{xy})$ as in-plane strains and stresses, respectively and $(\epsilon_z, \epsilon_{zx}$ and $\epsilon_{zy})$ and $(\sigma_z, \sigma_{zx}$ and $\sigma_{zy})$ as transverse strains and stresses, respectively. It may be noted here that in-plane strains are continuous across the interface while the corresponding stresses are discontinuous. Similarly transverse stresses are continuous across the interface while the corresponding strains are discontinuous across the interface. Generally it is difficult to achieve this feature in pure displacement- or stress-based models. We will see later that in the iterative model described later this feature has been realized.

Recognizing that the formulation of the governing equations of a structural element

Table 1. Nature of stress and strain components across the interfaces.

Continuous	ϵ_x	ϵ_y	ϵ_{xy}	σ_z	σ_{zx}	σ_{zy}
Discontinuous	σ_x	σ_y	σ_{xy}	ϵ_z	ϵ_{zx}	ϵ_{zy}

has a basis in the energy principles, it may be noted that the approach for modelling is essentially the Kantorovich form of the Rayleigh-Ritz method. Two classical approaches will be readily evident. In the first one the displacement is expressed in a series form in the thicknesswise or z -coordinate, leaving the rest in a functional form, and the governing equations are established retaining a few appropriate terms. We shall call such models here 'the displacement-based models'. The second approach treats stresses as primary variables. Soni & Pagano (1988) discuss an interesting new model based on this approach in this volume and the present discussion will be limited to displacement models.

2. Displacement-based models

The procedure consists of expanding the displacements in terms of the thicknesswise coordinate as,

$$\begin{aligned} U(x, y, z) &= \sum_{i=0,1,2} z^i u_i(x, y), \\ V(x, y, z) &= \sum_{i=0,1,2} z^i v_i(x, y), \\ W(x, y, z) &= \sum_{i=0,1,2} z^i w_i(x, y). \end{aligned} \quad (1)$$

Retaining a few terms and utilizing the energy principle or direct equilibrium considerations, the governing equations and boundary conditions are established (Reissner & Stavsky 1961; Dong *et al* 1962; Yang *et al* 1966; Whitney & Leissa 1969; Whitney & Pagano 1970; Whitney & Sun 1973; Nelson & Lorch 1974; Lo *et al* 1977). Models based on (1) have no provision for satisfaction of the plate surface boundary conditions. In general, surfaces of the laminate may receive both normal and tangential loads. A general method for modelling plates satisfying the plate surface conditions, as applied to isotropic plates is discussed by Vijaya Kumar & Krishna Murty (1988a). In this study Lagrangian multipliers were used to satisfy the normal stress condition at the plate surface. The results did not indicate significant improvement in the accuracies in predicting stresses, commensurate with the complexity of the formulation. In practice plates are usually subjected to normal surface loads only, and in that case, shear strains at plate surfaces are zero. This feature can be incorporated into the models much more easily by rewriting the expansion for displacements in the form,

$$\begin{aligned} U(x, y, z) &= \sum_{i=0,1,2} p_i(\xi) u_i(x, y), \\ V(x, y, z) &= \sum_{i=0,1,2} p_i(\xi) v_i(x, y), \\ W(x, y, z) &= \sum_{i=0,1,2} q_i(\xi) w_i(x, y), \end{aligned} \quad (2)$$

where

$$\begin{aligned} p_0 &= 1, \quad q_0 = 1, \quad p_1 = \xi, \quad q_1 = \xi, \quad p_2 = \xi^2/2, \\ p_n &= \xi(1 - \xi^{n-1}/n), \quad n = 3, 5, 7, \dots, \\ q_n &= 1 - \xi^n, \quad n = 2, 4, 6, \dots, \\ p_n &= \xi^2(1 - 2\xi^{n-2}/n), \quad n = 4, 6, 8, \dots, \end{aligned}$$

$$q_n = \xi(1 - \xi^{n-1}), \quad n = 3, 5, 7, \dots, \quad (3a)$$

$$\left. \begin{aligned} u_1 &= -w_{0,x} \\ v_1 &= -w_{0,y} \end{aligned} \right\},$$

$$\left. \begin{aligned} u_2 &= -w_{1,x} \\ v_2 &= -w_{1,y} \end{aligned} \right\}, \quad (3b)$$

and

$$\xi = z/h. \quad (3c)$$

Note that u_1 and v_1 are related to w_0 as in (3b) to accommodate Kirchhoff's model as a special case. This represents a generalization of several models currently available in the literature (Murthy 1981; Bhimaraddi & Stevens 1984; Reddy 1984, 1987; Krishna Murty 1987, 1988; Krishna Murty & Harikumar 1988; Krishna Murty & Vellaichamy 1988b).

In the displacement-based models all stresses can be estimated using the constitutive relations. We shall call such estimates of stresses as "the direct estimates". However, since the material constants are discontinuous across the interfaces, direct estimates of all stresses will be discontinuous across the interface. Therefore it is difficult to realize the true nature of interlaminar stresses as they are not continuous across the interfaces (see table 1). Thus direct estimates of interlaminar stresses are unlikely to be accurate. On the other hand, since in-plane stresses σ_x , σ_y and σ_{xy} are discontinuous across interfaces, their direct estimates can be expected to be accurate. A $[0/90]_s$ laminated plate strip, infinitely long y -axis simply supported at opposite edges at $x = 0, a$, subjected to sinusoidal load

$$(\sigma_z)_{z=\pm h} = \pm q_0 \sin(\pi x/a)$$

was analysed in detail by Krishna Murty & Vellaichamy (1988b) using the higher order shear deformation theory (HOST) and the classical laminated plate theory (CLPT). All the four layers of the laminate have equal thickness and the total plate thickness is $2h$. The middle surface corresponds to $\xi = 0$, and $\xi = 0.5$ represents the interface. Material constants of the 0° layer are taken to be $E_L/E_T = 25$, $E_T/E_t = 1$, $G_{LT}/E_T = 0.5$, $G_{LT} = G_{Lt}$, $G_{Tt}/E_T = 0.2$, $\nu_{LT} = \nu_{Tt} = \nu_{tL} = 0.25$. A comparison of a typical in-plane stress normalized with the applied load $\sigma_x^* = \sigma_x/q_0$ is shown in table 2.

The exact solution is based on the theory of elasticity. It is clear that the σ_x^* is estimated fairly accurately in HOST and the discontinuity at the interface is revealed

Table 2. Comparison of σ_x^* ($2h/a = 0.08$).

ξ	Exact	Direct estimates	
		HOST	CLPT
0.2	1.46	1.52	1.83
0.4	2.93	3.13	3.67
0.5	3.67	4.00	4.59
	87.85	98.45	107.90
0.6	115.40	121.21	129.48
0.8	173.50	172.03	172.65
1.0	236.95	231.79	215.80

Table 3. Comparison of σ_z^* ($2h/a = 0.08$).

ξ	Exact	Direct estimate HOST	Statically equivalent estimate	
			HOST	CLPT
0.2	0.255	0.080	0.257	0.259
0.4	0.509	0.184	0.513	0.519
0.5	0.636	$\begin{cases} 0.252 \\ 0.504 \end{cases}$	0.640	0.646
0.6	0.754	0.647	0.758	0.763
0.8	0.932	1.001	0.934	0.936
1.0	1.000	1.475	1.00	1.0

satisfactorily. Further we see that the estimates by CLPT, contain a error of about 10% only in the maximum value of σ_x^* , even when the thickness to width ratio is as large as 0.08. Comparison of a typical interlaminar stress, $\sigma_z^* = \sigma_z/q_0$, with the exact solution is shown in table 3.

The direct estimates indicate an unrealistic discontinuity at the interface and contain an error of 47.5% in the maximum value of the normal stress at the plate surface. Recognising that the governing equations in these models represent elemental equilibrium in an average sense, it may be noted that the equations of equilibrium, representing pointwise equilibrium,

$$\sigma_{ij,j} = 0, \quad i, j = x, y, z, \quad (4)$$

are available to obtain better estimates of interlaminar stresses than the direct estimates.

Substituting the in-plane estimates for stresses σ_x , σ_y and σ_{xy} in the equations of equilibrium (4) and integrating, one can get estimates to interlaminar stresses as,

$$\begin{aligned} \sigma_{xz} &= - \int (\sigma_{x,x} + \sigma_{xy,y}) dz + \text{constant}, \\ \sigma_{yz} &= - \int (\sigma_{y,y} + \sigma_{xy,x}) dz + \text{constant}, \\ \sigma_z &= - \int (\sigma_{xz,x} + \sigma_{yz,y}) dz + \text{constant}. \end{aligned} \quad (5)$$

Such estimates are referred to here as “statically equivalent estimates”. We see from table 3 that such estimates for interlaminar stresses display the required continuity across the interface and agree closely with the exact solution. It may be noted that the statically equivalent estimates of interlaminar stresses by the classical plate theory are also close to the exact solution. Table 4 shows a comparison of the interlaminar strain,

$$\varepsilon_z^* = \varepsilon_z/\varepsilon_0,$$

where $\varepsilon_0 = (2/D_{11}\pi^2)(a/h)^2$ and D_{11} is the plate bending rigidity.

Direct estimates display an unrealistic continuity across the interface $\xi = 0.5$. The statically equivalent estimates of strain are obtained using the statically equivalent estimates of interlaminar stresses and direct estimates of in-plane stresses in the constitutive relations. These statically equivalent estimates of strains display the

Table 4. Comparison of ε_z^* .

ξ	Exact	Direct HOST	Statically equivalent estimate	
			HOST	CLPT
0.2	-0.013	-0.035	-0.015	-0.024
0.4	-0.027	-0.071	-0.032	-0.048
0.5	$\begin{cases} -0.034 \\ -0.058 \end{cases}$	-0.088	$\begin{cases} -0.043 \\ -0.074 \end{cases}$	$\begin{cases} -0.060 \\ -0.087 \end{cases}$
0.6	-0.086	-0.106	-0.094	-0.105
0.8	-0.151	-0.141	-0.149	-0.149
1.0	-0.236	-0.176	-0.228	-0.205

required discontinuity in strains at the interface and agree closely with the exact solution.

Thus we see, that by choosing statically equivalent estimates of interlaminar stresses and strains and direct estimates of in-plane stresses and strains, displacement-based models can be used to study stresses in laminates. Nevertheless from (5) it may be noted that in view of the availability of only a single constant of integration, the interlaminar stresses estimates may not, in general, satisfy plate boundary conditions at both the plate surfaces, $z = \pm h$. However, experience based on numerical studies (Krishna Murty & Vellaichamy 1988b) indicates that this violation is minimal and may not be of real practical consequence.

3. Iterative modelling

As an extension to 'displacement-based modelling', it is possible to construct a hierarchy of models, wherein the displacement field at a given stage of the iterative model is deduced from the statical equivalent strains corresponding to the previous step of iteration. Such models are referred to here as 'iterative models'. Further, the difficulty in satisfying the second plate surface boundary condition may also be removed by obtaining the basic displacement variables, $u_i, v_i, w_i \dots$ etc. as a solution of the differential equations representing the plate surface boundary conditions instead of the "elemental equilibrium equations", whose satisfaction is not in any way essential, since local equilibrium equations are used to obtain the interlaminar stresses. Recently Valisetty & Rehfield (1985) presented a comprehensive model wherein this new hypothesis, namely that statically equivalent stresses from classical theory can be used to obtain transverse shear and normal strains, was introduced in place of the traditional Kirchhoff's hypothesis of zero transverse shear and normal strains. Krishna Murty & Vijaya Kumar (1987) and Vijaya Kumar & Krishna Murty (1988b) introduced the idea of utilizing plate surface boundary conditions, as governing equations, instead of the 'elemental equilibrium equations'.

A comparison of the displacements and stresses in a $[0/90]_s$ laminated strip subjected to sinusoidal loading of $(\sigma_z)_{z=\pm h} = \pm q_0 \sin \pi x/a$ is given in table 5. Higher accuracies attained at the second stage of the iteration are evident from this comparison.

Table 5. Comparison of displacements and stresses.

Quantity	ξ	CLPT	HOST ¹	Iterative ²	Exact
W^*	0	1.0	1.51	1.56	1.56
	1	1.0	1.51	1.56	1.56
U^*	1	0.126	0.135	0.138	0.138
σ_{x/q_0}	0.5	$\begin{Bmatrix} 2.16 \\ 53.95 \end{Bmatrix}$	$\begin{Bmatrix} 2.00 \\ 49.22 \end{Bmatrix}$	$\begin{Bmatrix} 1.82 \\ 44.11 \end{Bmatrix}$	$\begin{Bmatrix} 1.83 \\ 43.92 \end{Bmatrix}$
	1	107.9	115.9	118.7	118.5
σ_{xz/q_0}	0.5	5.09	5.05	5.02	5.02
σ_{z/q_0}	0.5	0.323	0.320	0.318	0.318

$W^* = W/W_{\text{CLPT}}$; $U^* = U/U_{\text{CLPT}}$; ¹Krishna Murty & Vellaichamy (1988b); ²Krishna Murty & Vijaya Kumar (1987).

4. Finite element modelling

The need for converting these models into a finite element form in order to be able to apply them to general laminates is obvious. In the displacement methods broadly two approaches may be considered for this purpose, namely (i) direct utilization of the three-dimensional finite elements, and (ii) finite elements based on classical laminated plate theory and higher order models. Isoparametric elasticity elements have been successfully used to study stresses in laminates (Whitcomb *et al* 1982; Carlsson 1983). Apart from the requirement of large degrees of freedom to model the necessary detail, it is observed (Whitcomb *et al* 1982) that the finite element solution is accurate except in one or two elements closest to the plane of discontinuity. The interface in a laminate is a plane of discontinuity in interlaminar strains. Further, isoparametric brick elements have no provision for inter-element stress continuity. Thus the suitability of such elements, for estimating interlaminar stresses which are continuous across interfaces, needs to be examined carefully. On the other hand, finite elements based on CLPT or HOST are relatively simple, and the provision to realize the necessary continuity in stresses and strains at the interfaces can be incorporated. Recently Kant (Kant 1988; Kant & Pandya 1988) has developed finite elements based on higher order theories and demonstrated their application through several examples. It will be interesting to study the performance of such elements in estimating interlaminar stresses at free edges and rivet holes to bring out the adequacy or otherwise of such elements for critical applications involving the estimation of interlaminar stresses.

5. Conclusions

In this paper displacement-based modelling of laminated composite plates is reviewed. Iterative modelling appears to be the most promising approach for the analysis of laminates as it has the provision to achieve the necessary continuities and discontinuities in stresses and strains across the laminate interfaces. The first step of the iterative model corresponds to the classical laminate plate theory with a small modification, namely statically equivalent estimates of transverse normal and shear strain replace

Kirchhoff's assumption of zero normal and shear strains. Several finite elements currently available in the literature may be easily modified to represent the first step of the iterative model. A similar approach can be implemented with finite element analysis based on higher order models also.

This work has been supported by the Aeronautics Research and Development Board, Ministry of Defence, Government of India. Valuable discussions with Professor K Vijaya Kumar, in particular his contributions to iterative modelling, are gratefully acknowledged.

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Effects of transverse shear and rotatory inertia on large amplitude vibration of composite plates and shells

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Abstract. A review of research in the area of large amplitude vibration of composite plates and shells is presented in this paper. The main focus of this paper is on the effects of geometric nonlinearity, transverse shear deformation and rotatory inertia on the vibration behaviour of single-layered plates and shells. Recent advances made in these areas are reviewed. Some literature on laminated plates and shells made of filamentary composite material is also included. Particular attention is given to the recent developments in the analytical methods of solution. Recent research leading to advances in numerical techniques is also included in the present overview.

Keywords. Large amplitude; nonlinear transverse shear; rotatory inertia; composite plates; composite shells.

1. Introduction

Nonlinear or large amplitude vibration of plates and shells have been considered by numerous researchers world-wide during the last 25 years or so. Many excellent review papers covering different aspects of these research areas have been written by research experts. A brief overview and a list of these review papers are presented later on in this paper.

In dealing with nonlinear vibrations of plates and shells it is important to realize that nonlinearities can arise in many different ways. If the material stress-strain behaviour is nonlinear, then the generalized Hooke's Law is no more valid resulting in material or physical-type nonlinearity. Alternatively, the nonlinearity may be due to the large deformations of an elastic body. This type of nonlinearity which is known as geometric nonlinearity may be due to stretching under large deformations or due to large curvature-displacement nonlinearity. The analytical and/or numerical solutions of these types of nonlinear problems are often very complex. This is mainly due to the fact that the advantages of uniqueness and superposition of solutions characteristic of problems governed by the linear differential equations do not exist in problems governed by the nonlinear governing differential equations. It is the purpose here to focus the attention on nonlinear vibration problems of composite moderately thick

plates and shells in which the geometric nonlinearities arise as a result of large deformations while the stress-strain laws are linear. In the case of moderately thick composite plates and shells, the effect of transverse shear deformation has been found to play a significant part in the large amplitude vibration behaviour. It is also clear from the open literature that the effect of rotatory inertia is much less important than the transverse shear deformation in the study of vibrations of plates and shells.

The use of filamentary composite materials in numerous applications has prompted many researchers to investigate the static and dynamic behaviour of structures made of such composite materials. In particular, the number of papers written in the area of plates and shells has increased considerably in the last 10 years. A composite plate or shell is often understood as being made of filamentary composite material, either in single layer or in multiple layers of usually equal thickness. Each layer is composed of fibres (such as glass, boron, graphite, kevlar) embedded in a matrix material such as epoxy resin. Fibre-reinforced composite laminates are now being used extensively in various engineering applications. They are particularly attractive due to the fact that structures made of composite materials exhibit a high strength-to-weight ratio and also provide enough flexibility to the designer to vary the material properties significantly by changing the fibre orientation as well as the stacking sequence of different layers of the composite laminated structure. In a typical single layer made of filamentary composite material, the fibre material usually has a much better elastic stiffness than the matrix material it is embedded in. Also for each layer of the composite material the values of Young's modulus parallel and normal to the fibres are substantially different resulting in greater stiffness and strength in the direction parallel to the fibres. Although every single layer made of filamentary composite material is truly nonhomogeneous, the assumption of homogeneity and anisotropy at each layer level is widely accepted in the solution of static and dynamic problems of composite structures.

Based on these definitions and assumptions, a review of the work done in the area of nonlinear vibrations of composite plates and shells is presented in this paper. Since the thickness has a pronounced effect on the behaviour of composite plates and shells, particular attention is given to the effect of the transverse shear deformation on the dynamic behaviour. An overview of advances made in both analytical and numerical methods of solution of single-layered composite plates and shells is presented with a brief review on laminated plates and shells.

2. Literature

Research carried out so far in the area of plates and shells indicates clearly that thickness plays a very important part in the static and dynamic behaviour of composite-material plates and shells. These effects are more pronounced in composite materials in comparison to isotropic materials. Such effects can be incorporated only by considering shear deformation theories. It should be noted here that classical thin plate and shell theories assume that fibres straight and normal to the undeformed median surface remain so after deformation. This assumption permits neglect of shear deformation effects in the analysis. It is well-documented in the literature that shear deformation theories predict slightly lower flexibilities of plates and shells resulting in an increase in the deflection and a decrease in the natural frequency as compared to the predictions of the corresponding classical theories. Therefore, it becomes necessary to

use shear deformation theories, particularly in high modulus composite plates and shells, for reliable prediction of static and dynamic behaviour. In addition, for nonlinear problems the effect of median surface stretching must be considered by accounting for nonlinear terms in the strain-displacement relations.

Research in the area of linear analysis of composite plates so far has produced numerous publications in the open literature. A number of shear deformation theories have been proposed and tested by several investigators. Stavsky (1965) proposed a shear deformation theory for laminated isotropic plates. Yang *et al* (1966) generalized this theory for laminated anisotropic plates by including the effects of shear deformation and rotatory inertia. The generalization is based on the Reissner-Mindlin plate theory applicable to homogeneous isotropic plates. There are other shear deformation theories such as the effective stiffness theory of Sun & Whitney (1973), the higher order theory of Whitney & Sun (1973) and the three-dimensional elasticity theory of Srinivas *et al* (1970). All these theories have been used by Pagano (1969, 1970), Whitney (1969), Whitney & Pagano (1970), Pagano & Hatfield (1972), Fortier & Rossettos (1973), Mau (1973), Sinha & Rath (1975) and Bert & Chen (1978) to study the linear static and dynamic behaviour of plates of mainly rectangular geometry with symmetric and antisymmetric cross-ply and angle-ply lamination configurations. A comparison of these theories for linear problems is reported by Bert (1974a, pp. 149–206). As a result of these studies it has been found that the shear deformation theory due to Yang *et al* (1966) is good enough for predicting the overall behaviour with respect to static as well as dynamic linear problems of laminated anisotropic plates. A review of the work done in this area using the finite element method is presented by Reddy (1981c) with several publications dealing with formulations and numerical results.

Nonlinear large deflection studies of plates accounting for the effect of the transverse shear deformation are few compared to linear plate problems. Noor & Hartley (1977, pp. 55–65) used the finite element method to include the effect of transverse shear strains. Reddy & Chao (1981b) developed a simple and efficient finite element based on the theory of Yang *et al* (1966) and used this element to study the nonlinear bending of composite plates. Wu & Vinson (1969) considered the effects of geometric nonlinearity, transverse shear deformation and rotatory inertia on the large amplitude vibration behaviour of rectangular plates using Berger's hypothesis. Sathyamoorthy & Chia (1980a) presented an improved version of the shear deformable theory for anisotropic single-layered plates and applied this theory to the large amplitude vibration of skew plates. Sathyamoorthy (1986c, 1987) has summarized the work in this area.

Zukas & Vinson (1971) and Dong & Tso (1972) considered the effect of transverse shear deformation, transverse isotropy and thermal expansion of layered orthotropic cylindrical shells. Whitney & Sun (1974) formulated the problem of laminated cylindrical shells to include the effects of transverse shear deformation and transverse normal strains. Refined theories for nonhomogeneous anisotropic cylindrical shells were developed by Widera & Logan (1980). Finite element analyses of layered anisotropic composite shells have been presented by Hsu *et al* (1981) and Noor & Mathers (1984) in which they have accounted for the transverse shear deformation. Further work on shear deformation linear theory of shells is given by Dong & Tso (1972), Bert (1974b), Cohen (1981), Shirakawa (1982, 1983), Soedel (1982), Jones & De Oliveira (1983), Sadasiva Rao (1983), Sharma & Darvirez (1985) and Sharma & Dimakakos (1985). In a review paper Reddy (1981c) has discussed the developments made in this area using the finite element methods.

Geometrically nonlinear static and dynamic analyses of shells of various shapes have been considered by some investigators in the recent past. First-order shell theories have been developed by Sanders (1959), Morley (1959) and several others. These theories have been applied to find solutions to shell problems. Comparisons indicate that these first-order theories are deficient in predicting stress singularities and higher natural frequencies. Reddy (1981d) has developed a shear-flexible finite element based on a shell theory that combines various first-order shell theories. Bhimaraddi (1984) and Liu & Reddy (1986b, pp. 288–294) have presented shell theories based on higher-order shear deformation theories. Static and dynamic problems of circular cylindrical shells have been considered using the analytical and finite element techniques. Stein (1986) has derived nonlinear equations of equilibrium that include the effects of transverse shearing of laminated thick plates and shells. He has also presented numerical results for buckling of thick rectangular plates in longitudinal compression.

3. Nonlinearity due to large deflection

In dealing with large amplitude vibrations of deformable bodies, the relationship between the extensional strains and the shear strains on the one hand and the displacement components on the other are taken to be nonlinear resulting in nonlinear strain-displacement relations. This is the type of nonlinearity most commonly treated in the literature. As a direct consequence of the nonlinear strain-displacement relations, the governing differential equations will turn out to be nonlinear. This is true in spite of the fact that the relationship between curvatures and displacement components are assumed to be linear. Another way of looking at this is to consider the total strain energy of the deformable elastic body which is the sum of the extensional or stretching energy and the bending energy. The extensional strain energy involves higher order nonlinear terms than quadratic in the normal displacement component whereas the bending strain energy remains quadratic in the displacement components. The three displacement components are the two in-plane displacements u^0 and v^0 and the normal or lateral displacement component designated usually by w . Thus, when these energy expressions are used to derive the equations of motion, they turn out to be a set of nonlinear governing differential equations.

In the case of plates, the nonlinear strain displacement relations leading to the geometrically nonlinear problems mostly discussed in the work are

$$\begin{aligned}\varepsilon_{\zeta}^0 &= cu_{,\zeta}^0 + sv_{,\zeta}^0 + (w, \zeta)^2/2, \\ \varepsilon_{\eta}^0 &= v_{,\eta}^0 + (w, \eta)^2/2, \\ \varepsilon_{\zeta\eta}^0 &= cu_{,\eta}^0 + sv_{,\eta}^0 + v_{,\zeta}^0 + w_{,\zeta}w_{,\eta}.\end{aligned}\tag{1}$$

In (1) ε_{ζ}^0 and ε_{η}^0 are the median surface normal strains and $\varepsilon_{\zeta\eta}^0$ is the in-plane shear strain. These relations are valid for a flat plate considering an ζ, η oblique coordinate system. θ is the skew angle such that $c = \cos \theta$ and $s = \sin \theta$. u^0 , v^0 and w are the displacement components of the mid-surface of the plate. The strains at a distance z measured normal to the median surface of the plate will then be

$$\begin{aligned}\varepsilon_{\zeta} &= \varepsilon_{\zeta}^0 + z\alpha_{,\zeta}, \\ \varepsilon_{\eta} &= \varepsilon_{\eta}^0 + z\beta_{,\eta},\end{aligned}$$

$$\begin{aligned}\varepsilon_z &= 0; \quad \varepsilon_{\zeta\eta} = \varepsilon_{\zeta\eta}^0 + z(\alpha_{,\eta} + \beta_{,\zeta}), \\ \varepsilon_{\zeta z} &= \alpha + w_{,\zeta}; \quad \varepsilon_{\eta z} = \beta + w_{,\eta}.\end{aligned}\quad (2)$$

It should be noted that (1) and (2) are nonlinear in the lateral displacement w but the curvature-displacement relationships remain linear. α and β are slope functions such that $\alpha = -w_{,\zeta}$ and $\beta = -w_{,\eta}$ for thin plates.

4. Governing equations for plates

Nonlinear or large amplitude vibrations of plates of various geometries have been studied extensively during the last few decades. For a background study on classical linear and nonlinear plate theories, the reader is referred to an excellent monograph by Leissa (1969). This monograph has a wealth of information on linear dynamic plate problems and provides additional information on geometric type nonlinearities. Sathyamoorthy & Pandalai (1972, 1973) presented a study of discs, membranes, rings, plates and shells by reviewing a total of 181 references and arrived at certain general conclusions regarding the nonlinear behaviour of these structural elements. The scope of the study was limited to those problems in which the geometric nonlinearities arise as a result of large deformations while the stress-strain laws are linear. Bert (1976a, b, 1979, 1980, pp. 207–258, 1982), Leissa (1977, 1978, 1981a, b, c, pp. 312–334, 1984, pp. 241–260), Reddy (1979, 1981c, 1982, 1985) and Sathyamoorthy (1983, 1986c, 1987) have discussed nonlinear problems of plates of various geometries, composite laminated plates and sandwich plates. The studies of Bert, Leissa and Sathyamoorthy largely deal with the advances made in the analytical methods of solution of plate problems. Reddy has reviewed the literature extensively to focus attention on the application of the finite element method to linear and nonlinear plate problems. The only book devoted entirely to the area of nonlinear analysis of plates (Chia 1980) deals with a variety of geometrically nonlinear static and dynamic problems. In addition, it gives a comprehensive list of papers published in this area until 1978. A limited amount of information on nonlinear plate vibrations is also available in Nayfeh & Mook (1979). All these references provide the reader with the necessary background material as well as information concerning advances made in several areas of plate vibrations. Recently, Sathyamoorthy (1986c) reviewed the literature published from 1983 to 1986 and noted an increased effort in the application of the finite element method to the solution of nonlinear static and dynamic plate problems.

The von Karman nonlinear plate theory can be generalized to include the effects of the transverse shear deformation and rotatory inertia for moderately thick plates. For the sake of generality, the dynamic plate equations applicable to a single-layered moderately thick anisotropic skew plate are presented here using oblique coordinates (Sathyamoorthy & Chia 1980a).

$$\begin{aligned}b_{22}F_{,\zeta\zeta\zeta\zeta} - 2b_{26}F_{,\zeta\zeta\zeta\eta} + (2b_{12} + b_{66})F_{,\zeta\zeta\eta\eta} \\ - 2b_{16}F_{,\zeta\eta\eta\eta} + b_{11}F_{,\eta\eta\eta\eta} = (w_{,\zeta\eta})^2 - w_{,\zeta\zeta}w_{,\eta\eta},\end{aligned}\quad (3)$$

$$L(J_1 + J_2) + M(w) = 0, \quad (4)$$

where,

$$J_1 = q(\zeta, \eta, t) - \rho h w_{,tt}, \quad (5)$$

$$J_2 = h(F_{,\eta\eta} w_{,\zeta\zeta} + F_{,\zeta\zeta} w_{,\eta\eta} - 2F_{,\zeta\eta} w_{,\zeta\eta}) \quad (6)$$

and the differential operators L and M are defined as

$$\begin{aligned} L = & r_1(\cdot)_{,\zeta\zeta\zeta\zeta} + r_2(\cdot)_{,\zeta\zeta\eta\eta} + r_3(\cdot)_{,\eta\eta\eta\eta} \\ & + r_4(\cdot)_{,\zeta\zeta\zeta\eta} + r_5(\cdot)_{,\zeta\eta\eta\eta} + r_6(\cdot)_{,\eta\eta\eta\eta} + r_7(\cdot)_{,\zeta\zeta\eta\eta} \\ & + r_8(\cdot)_{,\zeta\eta\eta\eta} + r_9(\cdot)_{,\eta\eta\eta\eta} + r_{10}(\cdot)_{,\zeta\zeta} + r_{11}(\cdot)_{,\eta\eta} \\ & + r_{12}(\cdot)_{,\zeta\eta} - r_{13}(\cdot)_{,\eta\eta} - 1, \end{aligned} \quad (7)$$

$$\begin{aligned} M = & N + e_9 R_i [(\cdot)_{,\eta\zeta\zeta} + (\cdot)_{,\eta\zeta\eta\eta}] + e_{13}(\cdot)_{,\zeta\zeta\eta\eta\eta\eta} \\ & + e_{14}(\cdot)_{,\zeta\zeta\zeta\zeta\eta\eta} + e_{15}(\cdot)_{,\zeta\zeta\zeta\eta\eta\eta} + e_9 R_i d_{15}(\cdot)_{,\zeta\zeta\eta\eta\eta\eta} \\ & + e_{16}(\cdot)_{,\zeta\eta\eta\eta\eta\eta} + e_{17}(\cdot)_{,\zeta\eta\eta\eta\eta\eta} + e_{18}(\cdot)_{,\eta\eta\eta\eta\eta\eta} \\ & + e_9 R_i d_7(\cdot)_{,\eta\eta\eta\eta\eta\eta}, \end{aligned} \quad (8)$$

with N given by

$$\begin{aligned} N = & e_1(\cdot)_{,\zeta\zeta\zeta\zeta} + e_{10}(\cdot)_{,\zeta\zeta\eta\eta} + e_{11}(\cdot)_{,\zeta\zeta\zeta\eta} + e_8(\cdot)_{,\eta\eta\eta\eta} \\ & + e_{12}(\cdot)_{,\zeta\eta\eta\eta} + e_{19}(\cdot)_{,\zeta\zeta\zeta\zeta\zeta} + e_{20}(\cdot)_{,\zeta\zeta\zeta\zeta\eta\eta} \\ & + e_{21}(\cdot)_{,\zeta\zeta\zeta\zeta\eta} + e_{22}(\cdot)_{,\zeta\zeta\zeta\eta\eta\eta} + e_{23}(\cdot)_{,\zeta\zeta\eta\eta\eta\eta} \\ & + e_{24}(\cdot)_{,\zeta\eta\eta\eta\eta\eta} + e_{25}(\cdot)_{,\eta\eta\eta\eta\eta\eta}. \end{aligned} \quad (9)$$

In (5) and (6), q is the lateral load applied per unit area of the plate, h is the uniform thickness, ρ is the mass density of the material of the plate and F is the stress function such that

$$N_\zeta = hF_{,\eta\eta}; \quad N_\eta = hF_{,\zeta\zeta}; \quad N_{\zeta\eta} = -hF_{,\zeta\eta}. \quad (10)$$

The coefficients b_{ij} , r_i and e_i in (3), (7), (8) and (9) are fully defined by Sathyamoorthy & Chia (1980a) in terms of the geometric and material parameters of the plate. In the case of anisotropic plates, the material parameters are written in terms of the orientation angle of the filaments ϕ , such that $m = \cos \phi$ and $n = \sin \phi$. They are also functions of E_L , E_T which are the major and minor Young's moduli, ν_{LT} , ν_{TL} which are the Poisson's ratios, and G_{LT} , G_{LZ} , G_{TZ} which are the shear moduli. It should be noted that the governing equations contain tracing constants T_s and R_i which represent the effects of transverse shear deformation and rotatory inertia, respectively.

The system of nonlinear differential equations (3) and (4) is of tenth order. Therefore five boundary conditions, as against four in the classical von Karman plate theory, are required in the present theory. The five boundary conditions along each edge consist of three corresponding to the out-of-plane conditions of the linear theory (with the thickness-shear flexibility taken into account) and two corresponding to the in-plane conditions of the nonlinear theory. These conditions which are obtained from the variational technique may be taken as a combination of the out-of-plane and in-plane conditions.

In the case of orthotropic plates, $\phi = 0$ and hence $m = 1$, $n = 0$. The corresponding material parameters can be derived from those applicable for anisotropic plates. For isotropic plates $m = 1$, $n = 0$, and $E_L = E_T = E$, $\nu_{LT} = \nu_{TL} = \nu$, $G_{LT} = G_{LZ} = G_{TZ} = G = E/2(1 + \nu)$.

Equations (3) and (4) are in terms of the stress function F and the normal

displacement w and therefore correspond to the stress function approach. Similar nonlinear dynamic equations can be derived in terms of the median surface displacements u^0, v^0 and w . Such equations are used in the displacement equations approach and can be readily found in the literature (Sathyamoorthy 1979, 1981a, b). When the effects of the transverse shear deformation and rotatory inertia are both neglected in the analysis, the corresponding tracing constants T_s and R_i are equated to zero. With appropriately simplified coefficients for the material constants, the governing equations (3) and (4) will reduce to those applicable for isotropic and orthotropic thin plates.

The nonlinear vibration of a circular plate has been solved by Sathyamoorthy (1982d) using the two different methods of solution, namely, the stress function approach and displacement equations approach. The two different methods given here lead independently to the stress function, F , and in-plane displacements, u^0 and v^0 , which are exact solutions to the corresponding governing equations. It is possible to obtain u^0 and v^0 from F and vice versa. The final results are independent of the method chosen to solve the problem. It has been found that the stress function approach has certain advantages over the displacement equations approach resulting in a substantial saving in analytical and computational efforts. The selection of this method will be of crucial importance in complex nonlinear problems such as those involving multiple modes or in cases where transverse shear and rotatory inertia effects are to be considered.

For a thin orthotropic plate of uniform thickness h with material axes of symmetry assumed to be parallel to the edges of the plate ($\phi = 0, m = 1, n = 0$), the differential equation governing the nonlinear flexural vibration of the plate may be readily derived from (3) and (4) in rectangular Cartesian coordinates. These equations in terms of nondimensional stress function f and lateral displacement W are

$$c_1 f_{,\zeta\zeta\zeta\zeta} + 2r^2 c_2 f_{,\zeta\zeta\eta\eta} + r^4 f_{,\eta\eta\eta\eta} = c_1 r^2 \{ (W_{,\zeta\eta})^2 - W_{,\zeta\zeta} W_{,\eta\eta} \}, \quad (11)$$

$$\begin{aligned} c_1 W_{,\zeta\zeta\zeta\zeta} + 2r^2 c_3 W_{,\zeta\zeta\eta\eta} + r^4 W_{,\eta\eta\eta\eta} + \mu r^4 W_{,\tau\tau} \\ = \mu r^2 (W_{,\zeta\zeta} f_{,\eta\eta} + W_{,\eta\eta} f_{,\zeta\zeta} - 2W_{,\zeta\eta} f_{,\zeta\eta}), \end{aligned} \quad (12)$$

where

$$\begin{aligned} \zeta = x/a, \eta = y/b, r = a/b, W = w/h, f = F/E_T h^3, \\ \tau = t(E_T h^2 / \rho b^4)^{1/2}, \mu = 12(1 - \nu_{LT} \nu_{TL}), c_1 = E_L / E_T, \\ c_2 = (E_T / G_{LT} - 2\nu_{LT})/2, c_3 = \nu_{LT} + \mu G_{LT} / 6E_T, \end{aligned} \quad (13)$$

a and b are lateral dimensions of the rectangular plate and F is the stress function.

Equations (11) and (12) are thin plate equations corresponding to the stress function approach. Equations corresponding to the displacement equations approach will be three in number, one each in terms of the displacement components u^0, v^0 and w of the mid-plane of the plate. In deriving the governing equations for moderately thick as well as thin plates, the effects of in-plane inertias have been ignored.

5. Shear effects on plates

Most of the work that has been done so far in the large amplitude vibration analysis of

moderately thick plates, single-layered or laminated, is based on the governing equations in terms of stress function F and lateral displacement w . Much of the recent analytical research on the effects of thickness shear flexibility and rotatory inertia has been done by Sathyamoorthy (1979, 1981a–c, d, pp. 367–368, e–g, 1982a, b, c, p. 5, 1984a–d, e, pp. 498–501, 1985a, b, pp. 301–307, c, pp. 634–642, d, pp. 483–484, 1986a–b), Sathyamoorthy & Chia (1980a–c, 1982), Celep (1980, 1982), Chia & Sathyamoorthy (1981), Sathyamoorthy & Prasad (1981, 1983, 1984), Dumir & Shingal (1985, 1986) and several others. The finite element method has also been used in recent years to solve this class of geometrically nonlinear plate problems (Kanaka Raju & Hinton 1980; Reddy & Chao 1981a, pp. 115–119, b, c; Reddy 1984, Di Sciuva 1986; Liu & Reddy 1986a, pp. 35–42, b, pp. 288–294). Moderately thick single-layered or laminated composite plates have been investigated for large amplitude vibration behaviour by Reddy (1981a, b), Reddy & Chao (1981a–c), Sathyamoorthy (1982c, p. 5, 1985c, pp. 634–642, 1986b), Sivakumaran & Chia (1985) and Liu & Reddy (1986a, b). Some recent investigations of Sathyamoorthy & Prasad (1983, 1984) are concerned with the influences of modal interaction on the nonlinear static and dynamic behaviour of moderately thick plates. Very little additional information is available on the effects of higher modes on the fundamental nonlinear frequencies of moderately thick plates. Chia (1980) and Yamaki (1981a) investigated this effect for thin plates and found that the effect of modal interaction was significant. In the case of moderately thick plates, a similar conclusion has been reached by Sathyamoorthy & Prasad (1983, 1984) in that the effect of coupling has been found to increase the frequency ratios for any given amplitude and is significant at moderately large amplitudes of vibration.

Analysis of flexural vibrations of elastic circular plates with initial imperfections is presented by Celep (1980, 1982). The analysis is applicable to the large amplitude vibration of moderately thick plates and includes the effects of transverse shear deformation and rotatory inertia. The classical nonlinear theory has been modified to include these effects as well as the effects of imperfections. Numerical results indicate that static and dynamic nonlinear behaviour are very much dependent upon the magnitude of the initial imperfections of the plate.

Moderately thick plates of various geometries have been treated in Sathyamoorthy (1979, 1981a–c, 1985a), Celep (1982), Reddy & Chao (1982), Chen & Lin (1984, pp. 567–576), Sivakumaran & Chia (1985) and others. A refined nonlinear theory to account for transverse shear deformation has been proposed by Reddy (1984). The study deals with the development of a higher-order theory, using parabolic distribution of the transverse displacement. The displacements along the in-plane directions are expanded as cubic functions of the thickness coordinate and the transverse deflection is assumed to be quadratic through the thickness. In dealing with moderately thick plates of various geometries, Sathyamoorthy (1981a, 1985a, etc.) made use of tracing constants to identify the effects of transverse shear deformation and rotatory inertia. This approach makes it very convenient to specialize the thick-plate equations for classical nonlinear thin plates. Sivakumaran (1985, pp. 1703–1710) has derived nonlinear governing equations, including the effects of transverse shear deformation and rotatory inertia, for free vibrations of interlaminated hybrid plates and has discussed numerical results presented in terms of variations of nonlinear frequencies with the amplitude of vibration.

6. Conclusions

The majority of the papers presented in this review paper deal with analytical and numerical methods used in the geometrically nonlinear dynamic analysis of plates and shells of various planforms. Due to space limitations the author has tried to review the plate theory completely while the presentation on shell theories is not complete. Based on the papers reviewed here it is clear that the effect of geometric nonlinearity on flat plates is one of hardening type, i.e., nonlinear frequency is dependent upon the amplitude of vibration and increases with it. Recent research activities continue to focus on the application of the finite element method to the nonlinear static and dynamic analysis of plates and shells. There is an increased interest in the investigation of several complicating effects on the large amplitude vibration of plates and shells and in the area of experimental research. Several papers are concerned with the development of new theories and new finite element models to include the effect of transverse shear deformation as well as to obtain improved solutions to dynamic plate problems. Very little information is found in the literature regarding the effects of higher modes on the nonlinear fundamental frequencies of plates and shells of various geometries and boundary conditions. Since composites are becoming more popular in high-performance design applications, there is real need for realistic theoretical and experimental prediction of the response of composite-material plates and shells. The material behaviour of these composite-material structural components is often not linear. The study of the dynamic behaviour of such components made of nonlinear materials deserves attention.

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Microstructures of directionally frozen *in situ* composites of multicomponent eutectics

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Abstract. Microstructural variations of binary (Sn-Pb, Sn-Cd, Pb-Cd), ternary (Sn-Pb-Cd) and quaternary (Sn-Pb-Cd-Zn) eutectics which were permitted to solidify directionally were investigated in terms of the controlled solidification rates. It was found that, at an appropriate freezing rate, the structure was lamellar and satisfied the Jackson-Hunt relationship, and furthermore, that the order of the layers present in the solidified eutectics was definite, such that in the case of Sn-Pb-Cd ternary eutectic alloy, for instance, the Sn-Cd layers would not be allowed to be in contact. An explanation for this could be the high interfacial energy between the two.

Keywords. Multicomponent eutectic; directional solidification; stacking sequence; interfacial energy; Sn-Pb-Cd-Zn alloy.

1. Introduction

Use of a unidirectional freezing technique has been wide-spread in obtaining mostly lamellar or needle-like structures in specific orientations. It has been widely accepted that the morphology of such an alloy can be plate-like, rod-like, globular, cellular or dendritic, depending upon the conditions adopted. The application of the directional solidification has been limited in most cases to binary eutectics and in a few cases to ternary eutectics. It will certainly be difficult to apply this technique to multicomponent eutectics to produce constituents in a desired morphology. Here, attempts are made to characterize the morphology of unidirectionally solidified multicomponent eutectics.

The materials chosen for this experiment were alloys of Sn-Pb-Cd ternary eutectics and Sn-Pb-Cd-Zn quaternary eutectics. In addition, binary eutectics of two out of three components, Sn, Pb and Cd, were examined as supplementary experiments.

2. Experimental methods

2.1 Specimen preparation

Specimens were prepared by melting tin, lead, cadmium and zinc each having purity greater than 99.9%. Eutectic alloys adopted for binary systems in this experiment are combinations such as Sn-Pb, Sn-Cd, Pb-Cd, and for ternary systems, Sn-Pb-Cd eutectics, and for quaternary systems, Sn-Pb-Cd-Zn eutectics.

Specimens having eutectic compositions were sealed in an evacuated silica tube with 8 mm inner diameter. The ultimate pressure obtained in this silica tube was of the order of 10^{-3} Pa. It had once been melted beforehand in order to assure compositional homogenization and, then, allowed to remelt and solidify unidirectionally by the Bridgman technique. The temperature gradients within the furnace at each eutectic temperature were 88 K/cm for all Sn-Pb, Sn-Cd and Pb-Cd systems, 30 K/cm for Sn-Pb-Cd and 37 K/cm for Sn-Pb-Cd-Zn systems. The size of the specimens was 8 mm (diameter) and 70 to 80 mm (length).

2.2 Microstructural observations

Microstructural observations were made both by optical and scanning electron microscopy. At first, the specimens were polished with emery paper, followed by cloth polishing in order to obtain a bright surface of the specimen. They were then etched with the suitable solution.

3. Results and discussions

The microstructure of binary eutectic alloys directionally frozen at various rates consists of a lamellar structure, the spacings of which vary with freezing rates. Figure 1 shows the relationship between lamellar spacings and freezing rates, from which it is obvious that the Jackson-Hunt relationship (Jackson & Hunt 1966) holds.

In the course of development of the theoretical treatment for the interlamellar spacings and the rate of solidification based on the volume diffusion as the governing step, Jackson & Hunt (1966) and Chadwick *et al* (1963–64) derived the expression describing the interfacial energy of the frozen phase in contact:

$$\sigma_{\alpha\beta} = [\gamma \cdot A(C_E - C_\alpha)L_E \cdot \rho_E] / [8 \cdot T_E \cdot D(1/m_\beta - 1/m_\alpha)], \quad (1)$$

where each of the symbols represents as follows:

$\sigma_{\alpha\beta}$: interface boundary energy, γ : arbitrary geometrical factor which may be equal to unity, C_E , C_α : composition (wt.%) of α -phase in eutectics, L_E , ρ_E and T_E : latent heat, density and temperature at eutectic composition, D : diffusivity of solute in the liquid. m_α , m_β : slopes of liquidus lines for α , β phases and A is a Jackson–Hunt constant. Adopting the values for corresponding letters for α , β phases for each alloy system as shown in table 1, the interphase boundary energies of the three binary eutectic systems investigated are computed as shown in table 2. It is clear that the interphase boundary energy between Sn-Cd phases is the greatest of the three combinations.

Figure 2 shows the microstructural variation of Sn-32Pb-18Cd ternary eutectics which have been allowed to solidify at various rates. The figures on the left and right (of figure 2) correspond, respectively, to the structure of the longitudinal and transverse

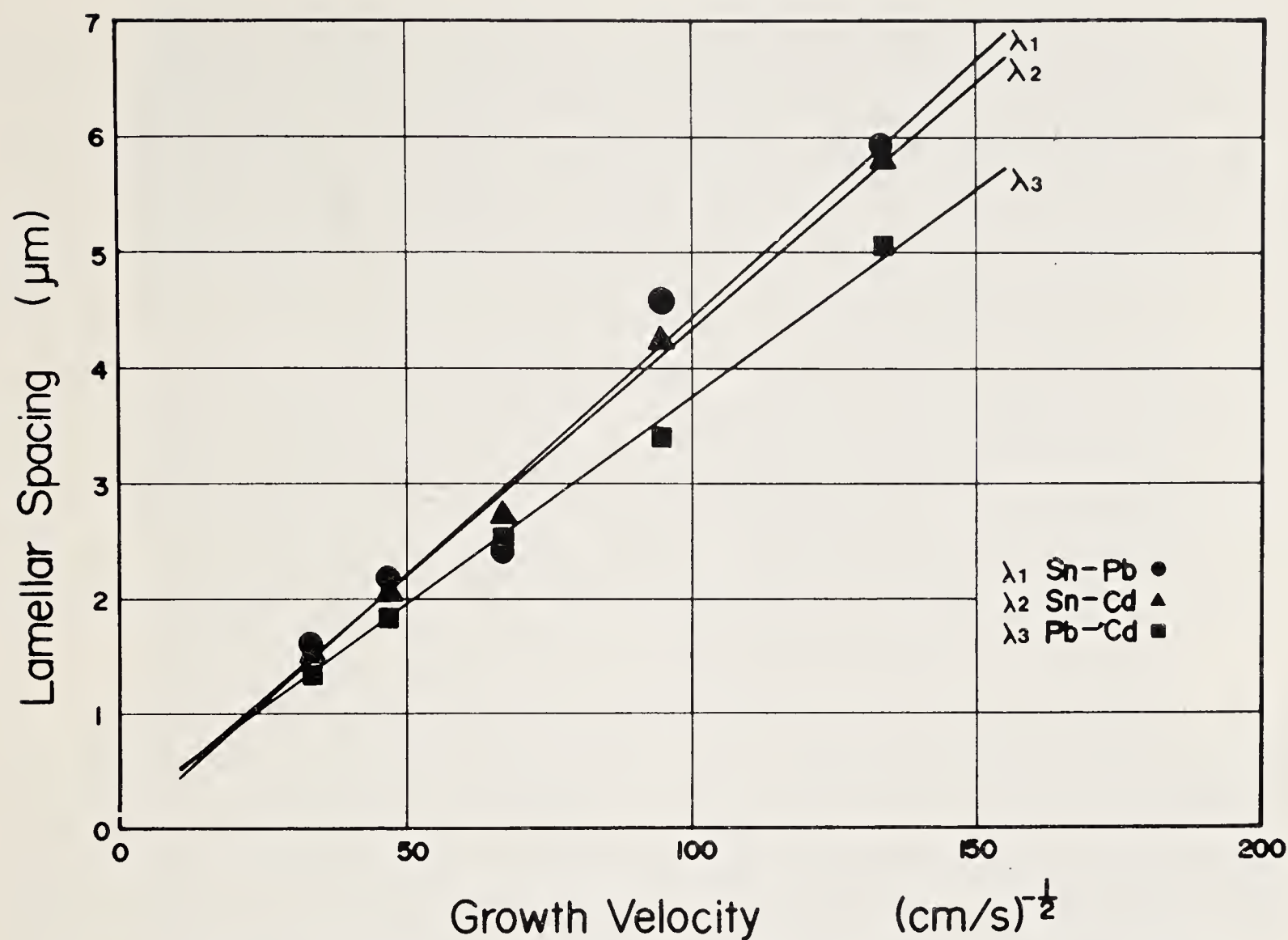


Figure 1. Logarithmic plot of lamellar spacing vs. growth rate for each binary eutectic alloy.

Table 1. Values used for estimating the interfacial energy between the phases.

System Alloy	A^2 ($\text{cm}^3/\text{s} \times 10^{-11}$)	C_E (wt.%)	C_α (wt.%)	L_E (cal/g)	ρ_E (g/cm^3)	T_E (K)	m_α ($^\circ\text{C}/\text{wt.}\%$)	m_β ($^\circ\text{C}/\text{wt.}\%$)
Sn-Pb	2.03	38.10	2.5	10.9	8.45	456	-0.83	3.20
Sn-Cd	1.88	32.25	5.2	13.62	7.68	450	-1.03	1.61
Pb-Cd	1.28	17.40	3.3	7.3	10.70	521	-1.10	4.80

Table 2. Calculated values of the interphase-boundary energies of the three eutectic systems investigated ($D = 2 \times 10^{-5} \text{ cm}^2/\text{s}$).

System	Sn-Pb	Sn-Cd	Pb-Cd
$\sigma_{\alpha\beta}$ ergs/cm ²	25.1	48.3	6.3

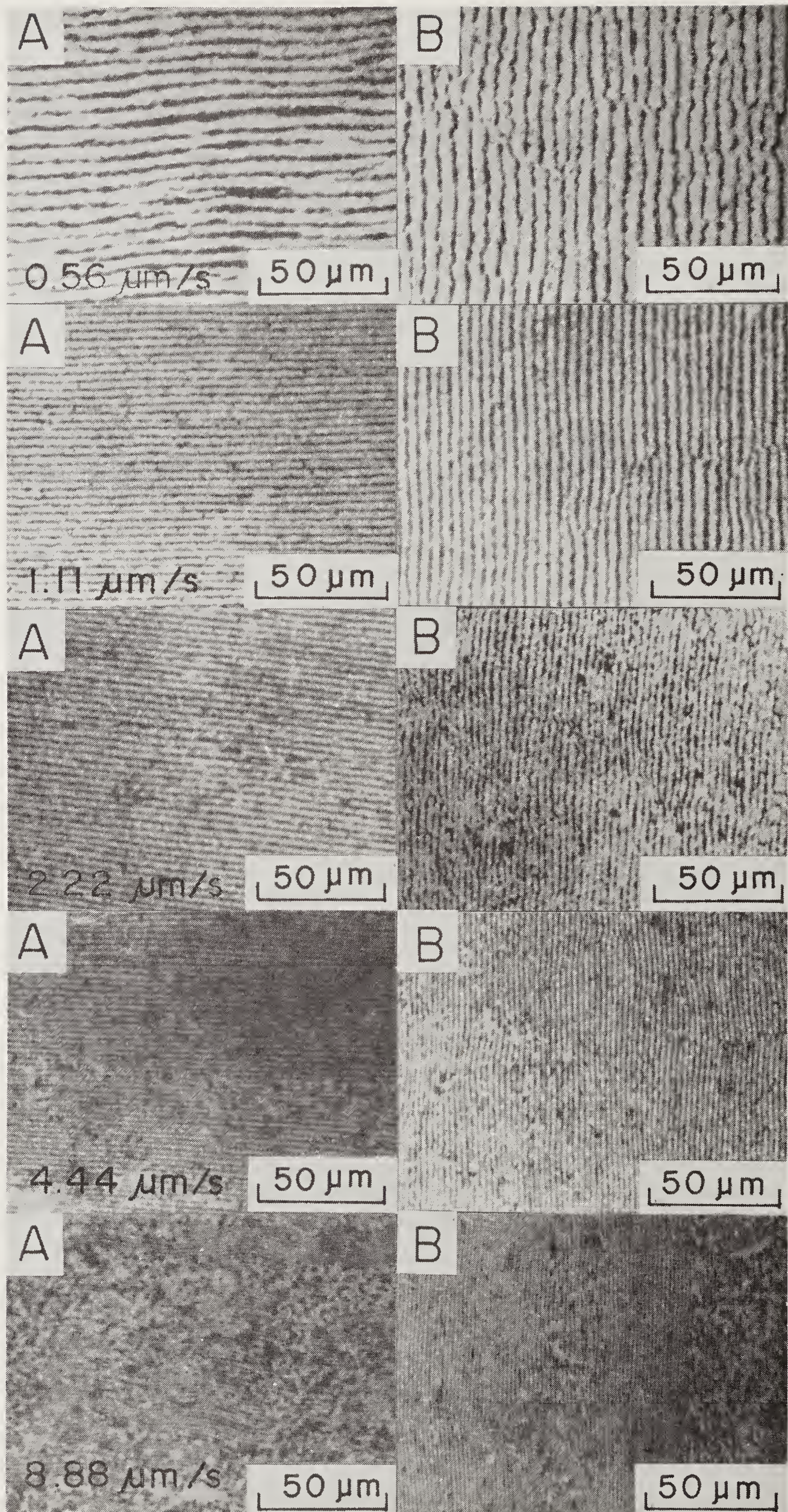


Figure 2. Optical microstructure at different growth velocities in Sn-Pb-Cd ternary eutectic alloy, (A) longitudinal section, (B) transverse section.

sections of the specimens. Apparently, the structural defects are the least in the specimens drawn at rates ranging from $1.11 \mu\text{m/s}$ to $4.44 \mu\text{m/s}$ and their columnar structure is in a state of ordered parallelism. But specimens which are allowed to solidify at the rate beyond or below this range have not only greater density of defects but also less ordered parallelism of lamellae. Obviously, it is seen that interlamellar spacing is smaller in rapidly solidified eutectics. The overall characteristics of the microstructure of the ternary alloy is similar to those of binary eutectics.

Figure 3 shows the various defects called (a) terminations appearing on the transverse section, (b) colony structure with illfitting boundaries, and (c) dendritic structure, both (b) and (c) appearing on the longitudinal sections. The defect (c) has often been seen in the portion where the composition is not that of the eutectic point. Figure 4 shows the scanning electron micrograph of the Sn-Pb-Cd ternary eutectic alloy where three phases are seen in parallel. Lineal analysis by X-ray indicates that the stacking sequence of the three layers Sn, Pb and Cd is specifically ordered such that no

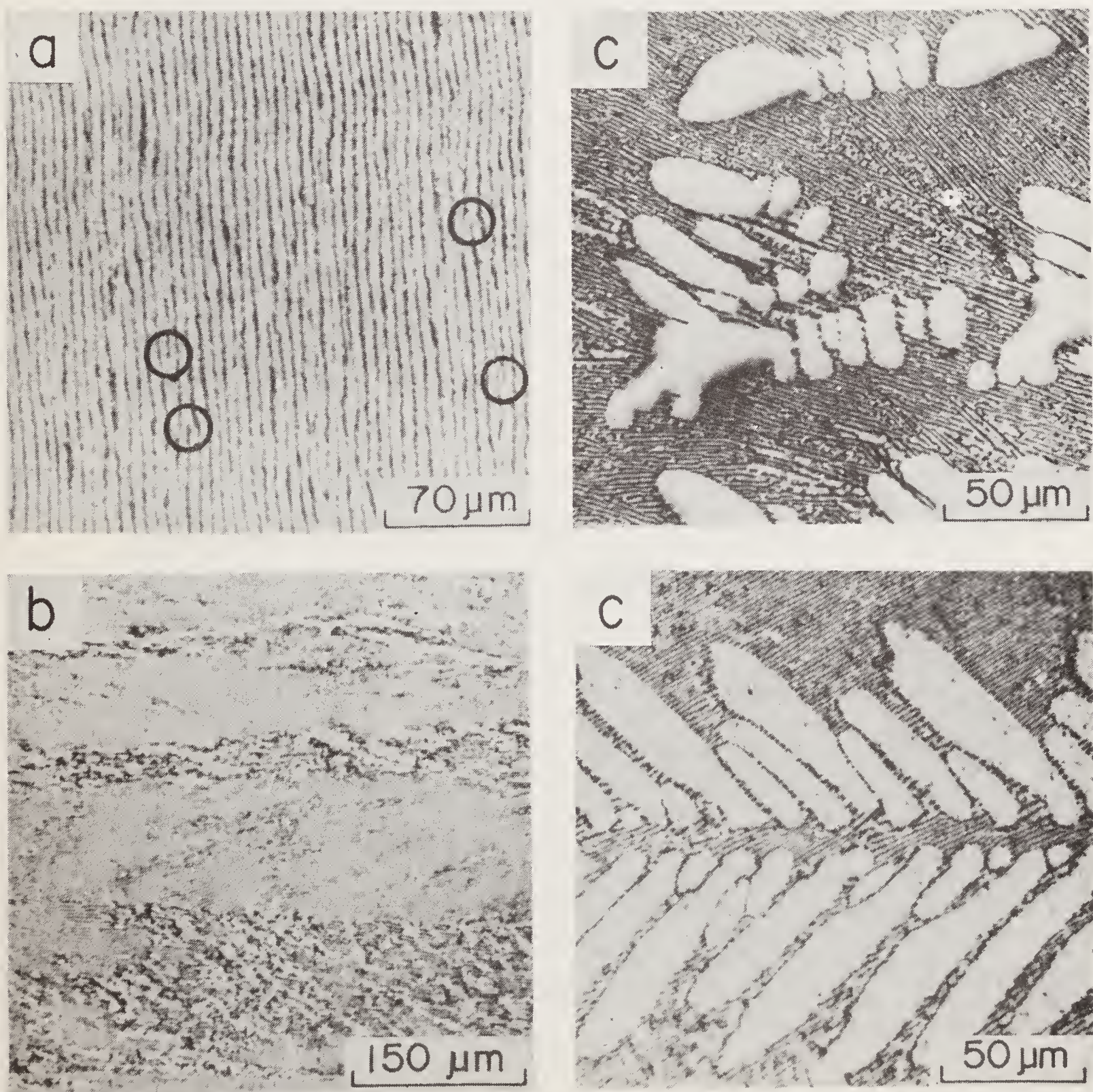


Figure 3. The observed defect structure of Sn-Pb-Cd ternary eutectic (a) termination, (b) colony structure, and (c) dendritic structure.

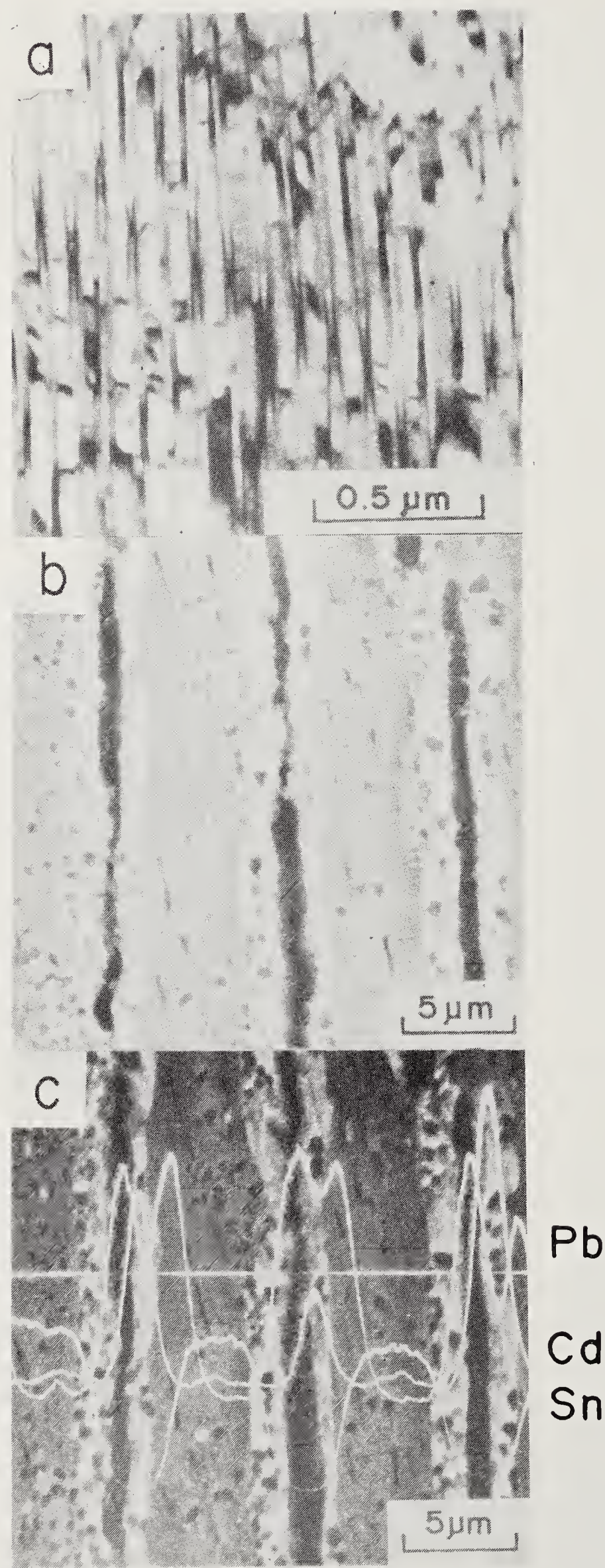


Figure 4. Lineal analysis by X-ray from a transverse section of Sn-Pb-Cd ternary eutectic alloy.

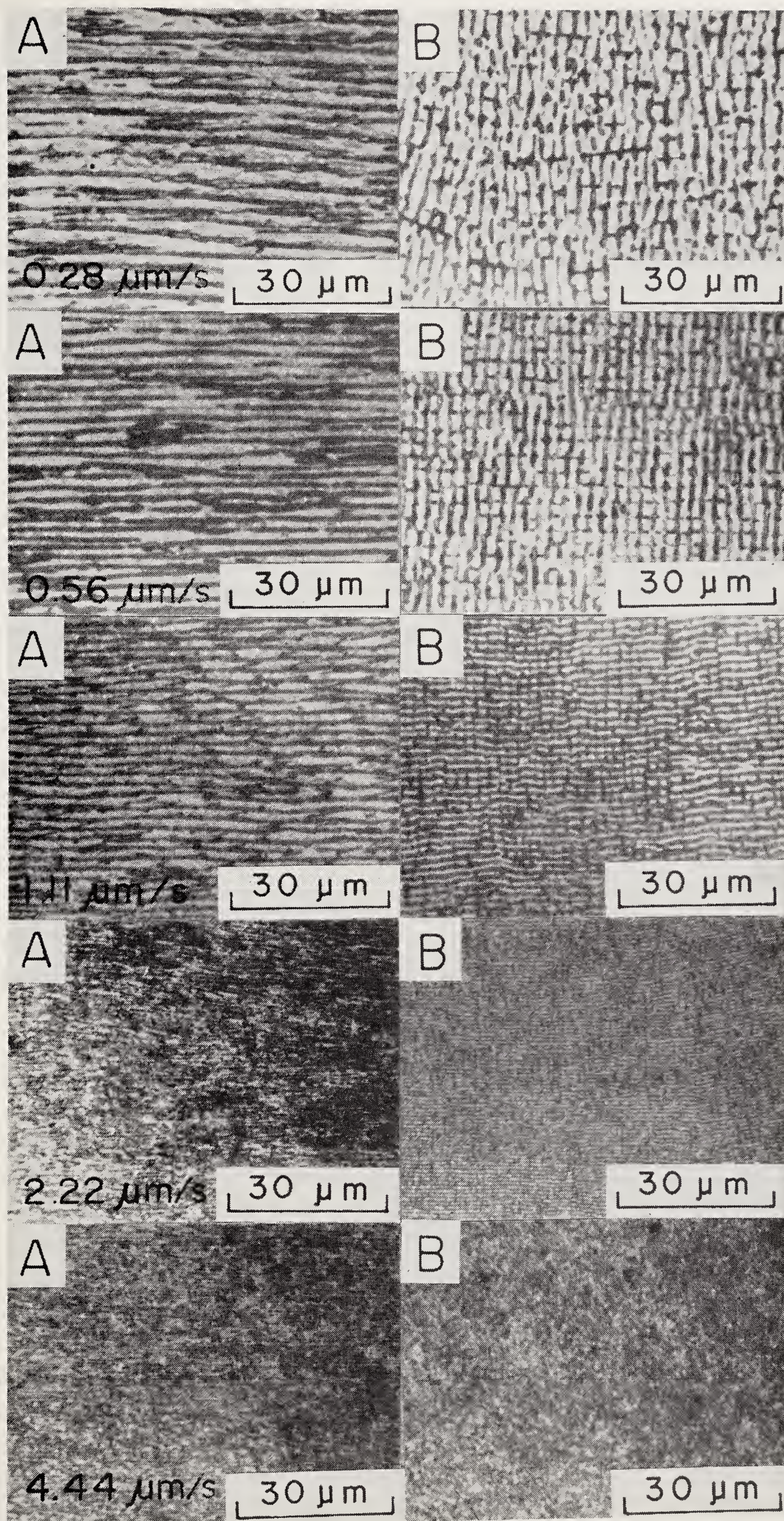


Figure 5. Optical microstructure at different growth rates in Sn-Pb-Cd-Zn quaternary eutectics, (A) longitudinal section, (B) transverse section.

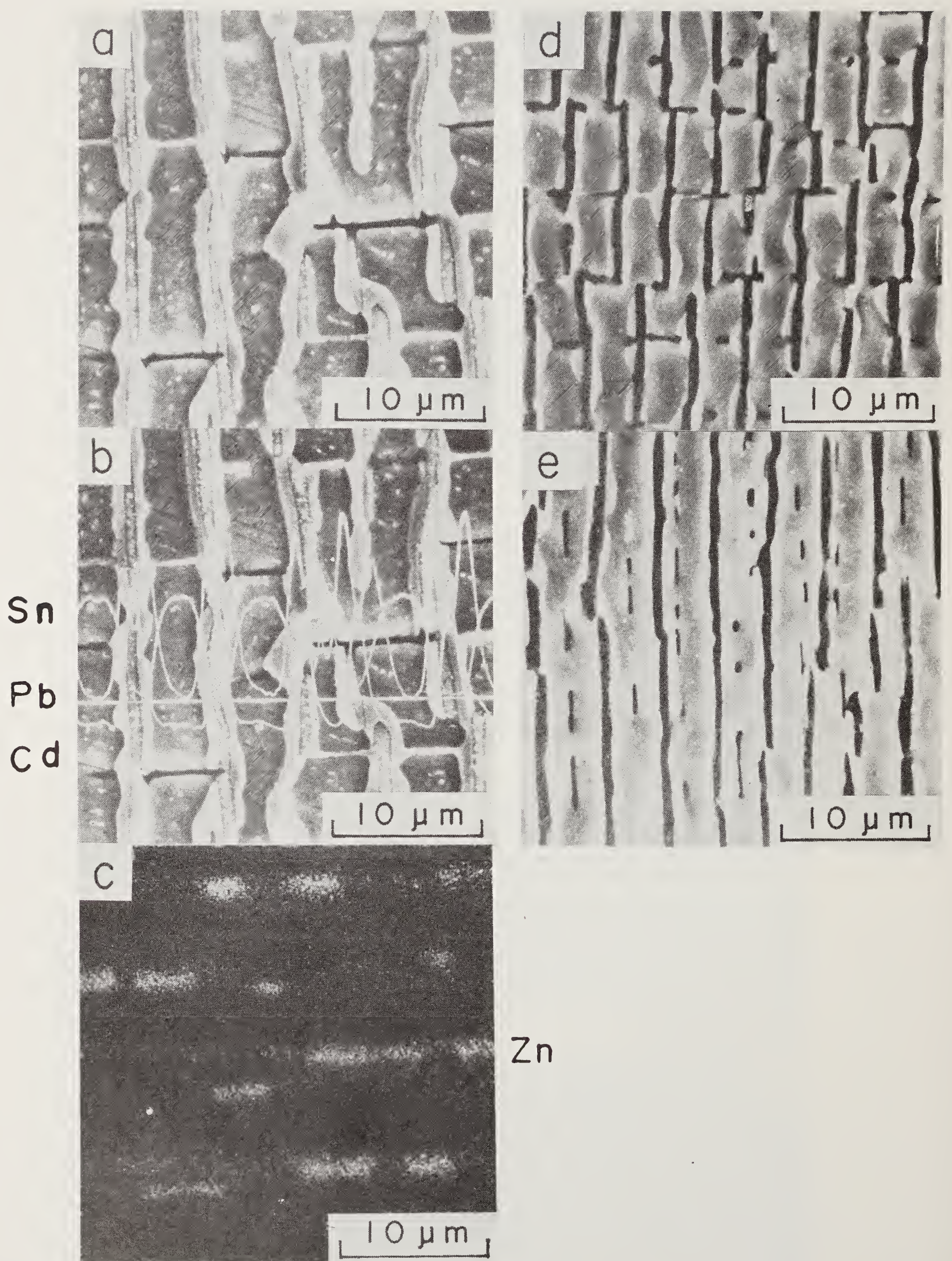


Figure 6. Scanning electron micrographs of quaternary Sn-Pb-Cd-Zn eutectics. (a) and (b) show the two portions with different morphologies in the four phases in the same specimen ($V = 4.44 \mu\text{m/s}$). The three phases Sn-Pb-Cd are oriented in parallel in the same order as in the ternary eutectic but the remaining Zn lies normal to the above three in (c) and parallel to them in (d) and (e).

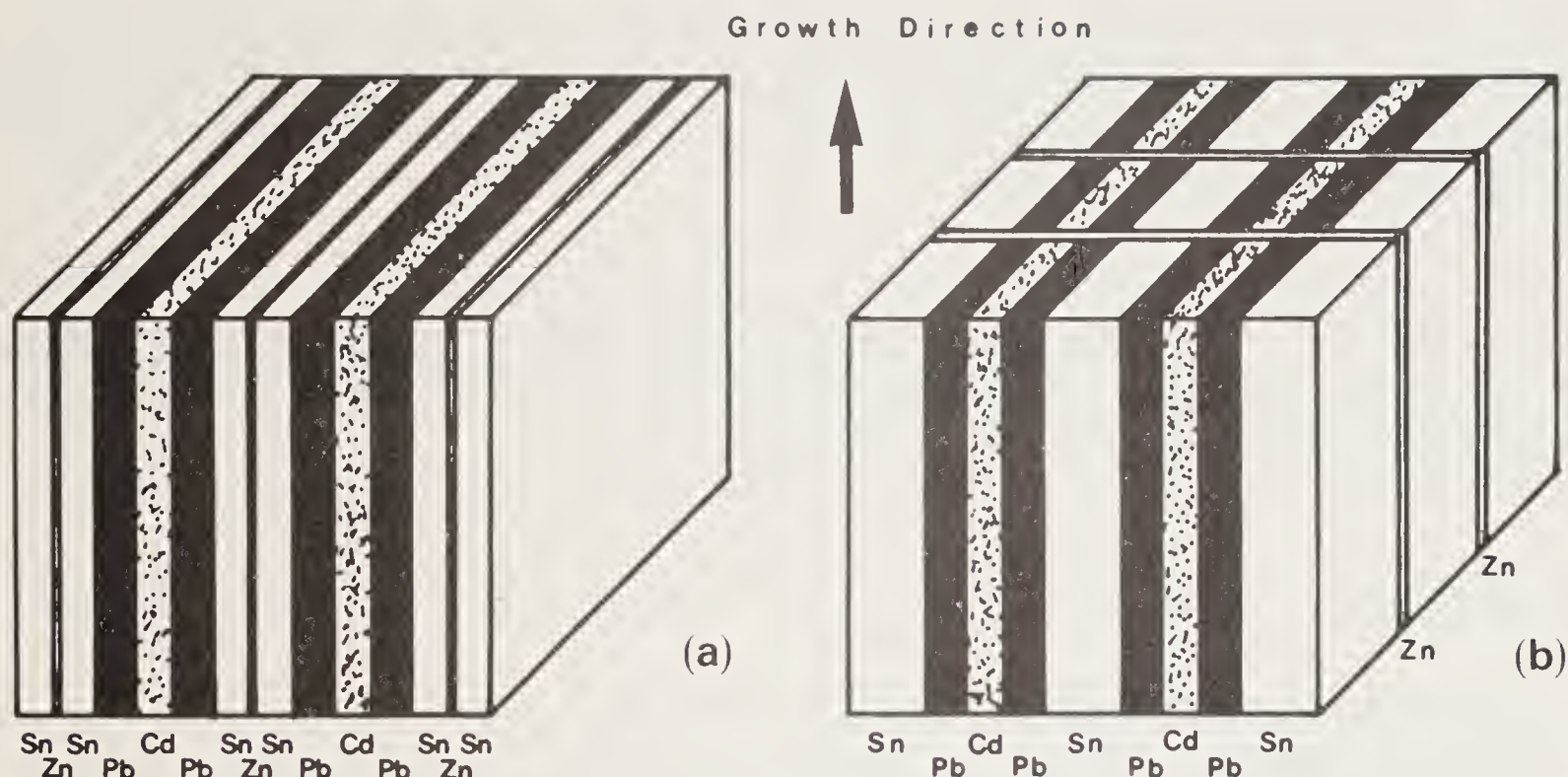


Figure 7. Illustration of the sequence of the four phases observed in quaternary plate like eutectic structures with the Zn layer in (a) parallel orientation, (b) normal orientation.

direct contact between Sn and Cd phases can occur. This leads to the assumption that the interfacial energy between Sn and Cd phases is excessively great, and that the formation of such a high energy boundary has been avoided by nature. This is in tentative agreement with the observations made here in the binary eutectics, provided the ternary eutectics have essentially the same composition as the binary ones. The solubility of one element into the other two among Sn, Pb and Cd is by no means excessive.

Figure 5 shows the microstructural variation of the quaternary Sn-28.6 wt% Pb-16.7 wt% Cd-2.25 wt% Zn eutectic alloy which has been solidified at various rates. Pictures in the (A) row correspond to the structure on the longitudinal section and in the (B) row to the structure on the transverse section. As before, the interlamellar spacings decrease as the rate of solidification is increased. The terminations can be seen more often in this system than in the rest of the eutectic alloys. The well-aligned structure is obtained at a rate ranging from 0.56 to 2.22 $\mu\text{m/s}$. At rates beyond 4.44 $\mu\text{m/s}$, its microstructure deteriorates in the sense that the plate-like morphology is not only bent but also broken.

Examinations by X-ray microanalyser demonstrate that there are two types of portions having different morphologies in the four phases. Figures 6a and b shows these two areas observed in the same specimen ($V = 4.44 \mu\text{m/s}$). The three phases Sn-Pb-Cd are oriented in parallel in the same order as in the ternary eutectic but the remaining Zn phase lies normal to the above three in one case and lies parallel to the above three in the other, being embedded within the Sn-phase with short yet elongated rod-like shape. Figure 7 shows schematically the sequence of the four phases observed, as analysed by the scanning electron microscope. Type (B) of figure 7 has been observed by other investigators (Tiller 1958) in the same alloy system but type (A) has seldom been found. In the case of quaternary eutectics, the order of the three layers has been similar to those of in the ternary one in that the contact of Sn and Cd layers does not occur.

The mechanisms of formation of the entirely different morphologies of the Zn phase must have been different and must have something to do with the mechanisms of solidification during which the solute atoms, in particular Zn atoms, have drifted. But it

is uncertain whether the Zn-phase forms the least interfacial energy boundary with the Sn-phase and forms high energy boundaries with the rest of the three. When the Zn phase is formed with perpendicular orientation, the contact of Zn with all the rest of the three phases can not be avoided.

The authors would like to express their sincere appreciation to Mita Industries Co., Ltd. for financial support in this investigation.

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Anisotropy of elastic constants of *in situ* Pb-Sn eutectic composite measured by pulse-echo method

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Abstract. Anisotropic elastic constants of Pb-Sn eutectic composites with layered structure have been measured by the pulse-echo method. The magnitude of the elastic constants was found to vary considerably depending upon the crystallographic directions as well as the directions relative to the plate-like eutectic constituents. The elastic constants of Pb-Sn eutectic bicrystal were determined taking the x_1 and x_3 axes, respectively, as the directions parallel to and normal to the growth direction of the eutectics. The interfacial energy of Pb-Sn layers was also determined. In addition, the anisotropy of the eutectics was confirmed by resistivity measurements.

Keywords. Anisotropic elastic constants; pulse-echo method; Pb-Sn eutectic bicrystal; *in situ* Pb-Sn eutectic composite.

1. Introduction

One of the methods of producing composite materials is to induce unidirectional solidification under appropriate conditions producing a regularly and alternatively stacked hard phase embedded in a soft matrix. Such materials are often called *in situ* composite materials and they are generally far from isotropic in their physical or mechanical properties because their microstructure is in most cases lamellar, needle-like or globular depending upon the solidification parameters such as the rate of solidification, the temperature gradient of the furnace used while cooling and the level of impurities accumulated at the advancing solid-liquid interface. The attendant microstructure obtained under specific conditions plays a critical role in the resultant anisotropy. This investigation is concerned with the determination of the anisotropic elastic constants of directionally solidified binary Pb-Sn eutectics with the given crystallographic orientation. The anisotropic nature of the composite is also confirmed by resistivity measurements.

2. Experimental method

2.1 Preparation of eutectic Pb-Sn single crystal

A mixture of Pb and Sn of 99.9% purity having the eutectic composition was melted

and then allowed to solidify unidirectionally. The growth of each phase was carried out by the melt-back technique as adopted by Canter & Chadwick (Chadwick 1963; Canter & Chadwick 1975). The mixture had been encapsulated within a quartz tube evacuated down to a pressure of 10^{-3} Pa before it was melted, so that the chemical composition of a sample was precisely at the eutectic point. The first melting and solidification process was carried out from one end of the capsule to the other and the second process was performed in the backward direction. This process was repeated several times. The sample was taken from the central part of an ingot, both ends of which had been removed in order to assure the homogenization of the structure. The rate of solidification adopted was 12 mm/hr and the temperature gradient of the furnace was 343 ± 2 K/cm at the melting point of Pb-Sn eutectics. The solid solution of α Sn containing 1.15 at.% Pb was also made by a similar method with the rate and gradient being the same. The microstructural observations were made using optical and transmission electron microscopes. Measurements of sound velocities in samples were carried out by use of the transducers PbTiO_3 and quartz, respectively, for emitting longitudinal and transverse waves.

Resistivity was measured by means of the four-probe method using a potentiometer.

3. Results and discussions

3.1 Microstructure and crystallography

Figure 1 shows the progressive grain growth of Pb-Sn eutectic constituents with increasing repetition of the unidirectional solidification in the to and fro directions. It is quite noticeable that the melt-back technique using the Bridgman method is quite a feasible method for growing large crystals. The crystallographic orientation relationship between the pertinent phases was assumed to be as follows from the results of previous workers:

$$\begin{aligned} \text{Growth direction} & \quad //(\bar{1}30)_{\text{Sn}}//(\bar{1}\bar{1}4)_{\text{Pb}}, \\ \text{Interfacial plane} & \quad //(\bar{3}\bar{1}0)_{\text{Sn}}//(\bar{2}\bar{2}1)_{\text{Pb}}, \end{aligned}$$

which have been reported by Verhoeven *et al* (1977) and Takahashi & Ashinuma (1958), both of whom adopted similar experimental conditions as in this investigation, although different growth rates were found to result in different crystallographic orientation relationships. This is schematically illustrated in table 1.

3.2 Density of α -Sn and Pb-Sn eutectics

The density of each constituent which is needed to calculate the elastic constant of this eutectic material was calculated from the lattice parameters following Vegard's law. It was estimated as follows:

$$\begin{aligned} \rho_{\alpha\text{-Sn}}(\text{Sn} - 1.15 \text{ at.\% Pb}) &= 7.35 \text{ g/cm}^3, \\ \rho_{\text{eutectic}} &= 8.36 \text{ g/cm}^3. \end{aligned}$$

3.3 Anisotropy of unidirectionally frozen Pb-Sn eutectics

Since the final purpose of our investigation is to determine the mechanical properties of

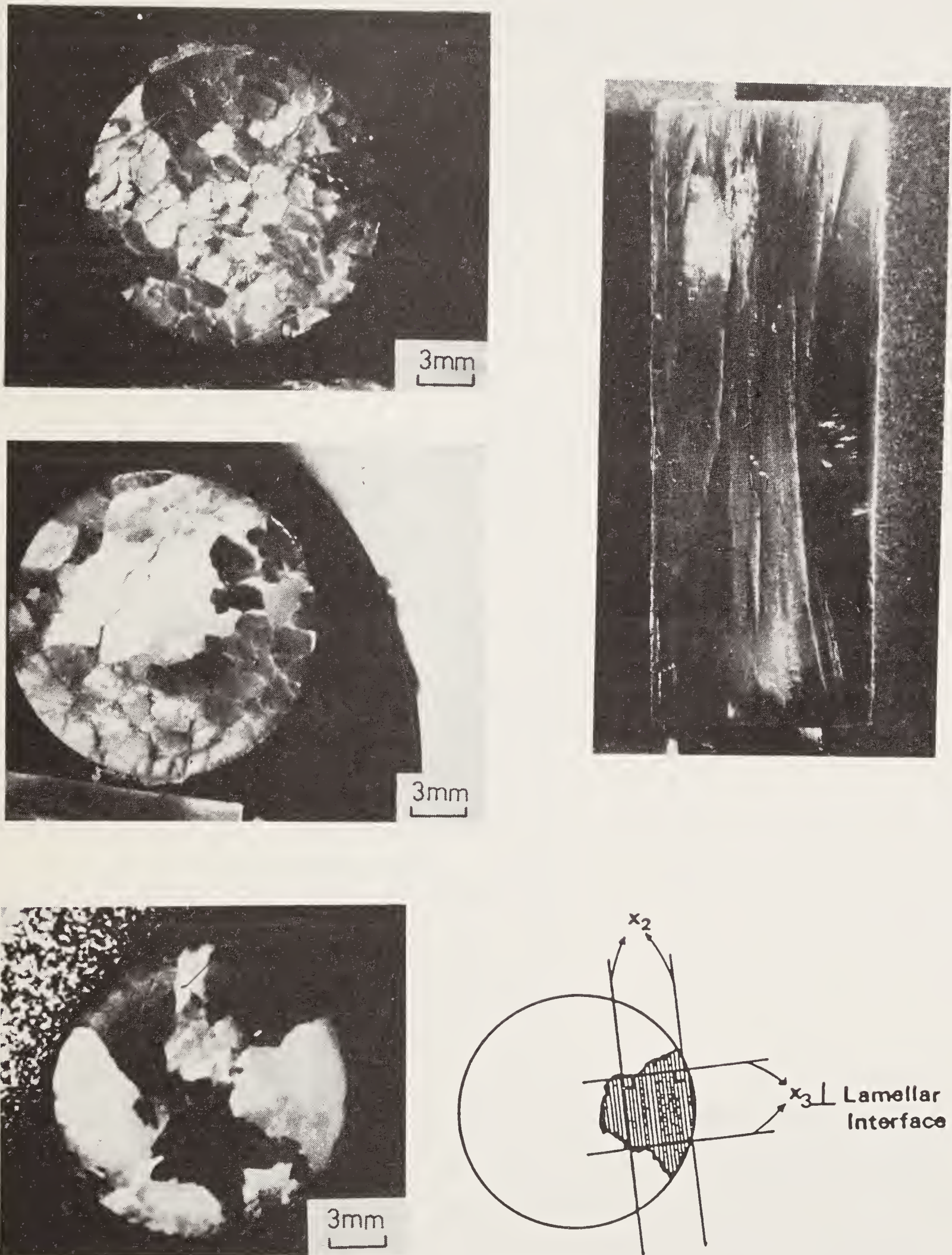


Figure 1. Grain growth by melt-back technique.

the frozen eutectics having the specific crystallography naturally chosen, it is considered appropriate to compare this result with those of the specifically oriented crystals obtained by other investigators. The elastic constants of tin and lead single crystals have been found for instance, by Mason (1976), Huntington (1958), Fedorov (1958) and Hearmon (1958) for Sn and by Huntington (1958), and Fedorov (1958), for Pb. Although some scatter is found in their data, in particular in the data

Table 1a. Effect of solidification rate upon crystallography of Pb-Sn eutectic alloys. Composition 38.1 wt.% Pb.

Rate (μ m/s)	Gradient (K/cm)	Crystallography
100	403	III
98	403	III
51	633	III, IV
51	633	III, IV
41	403	III, IV
41	403	II
31	633	I, II
21	403	II
21	403	I
10	403	I
5.1	403	I
2	403	I
2	633	I
1.3	633	I

Table 1b. Pb-Sn orientation relationship determined in this study.

Relationship	Interface, growth direction (GD) and orientation relationship
I	Int. $\parallel (3\ 1\ 0)\text{Sn} \parallel (2\ 2\ 1)\text{Pb}$ GD $\parallel [1\ 3\ 0]\text{Sn} \parallel [1\ 1\ 4]\text{Pb}$
II	Int. $\parallel (5\ 1\ 2)\text{Sn} \parallel (1\ 2\ 1)\text{Pb}$ GD $\parallel [2\ 4\ 3]\text{Sn} \parallel [1\ 1\ 3]\text{Pb}$
III	Int. $\parallel (23, 16, 0)\text{Sn} \parallel (2\ 1\ 0)\text{Pb}$ GD $\parallel [16, 23, 0]\text{Sn} \parallel [0\ 0\ 1]\text{Pb}$ and Int. $\parallel (23, 16, 0)\text{Sn} \parallel (2\ 2\ 1)\text{Pb}$ GD $\parallel [16, 23, 0]\text{Sn} \parallel [1\ 1\ 4]\text{Pb}$
IV	Int. $\parallel (1\ 0\ 0)\text{Sn} \parallel (1\ 1\ 1)\text{Pb}$ GD $\parallel [0\ 0\ 1]\text{Sn} \parallel [0\ 1\ 1]\text{Pb}$

given by Fedorov (1958), the difference in the data given by the rest of the authors is within the range of 10% scatter.

Here, adopting the data by Mason (1976) for tin, and that by Huntington (1958) for Pb, an attempt has been made to transform their data into our crystallographic orientation so that direct comparison of our result with theirs can be made. In doing so, the results by Mason and Huntington were suitably transformed to the crystallography of our specimen. The elastic constants of the Pb-Sn eutectics from the data by others were transformed in the coordinate system shown in figure 2. The transformation of the coordinate system is given by the tensor notation as:

$$T'_{ij} = a_{ik}a_{jl}T_{kl}, \tag{1}$$

where T'_{ij} and T_{ij} are the tensor properties in the new and old coordinate system and a_{ik} etc. are the direction cosines. Then, the transformation of the elastic constants given by

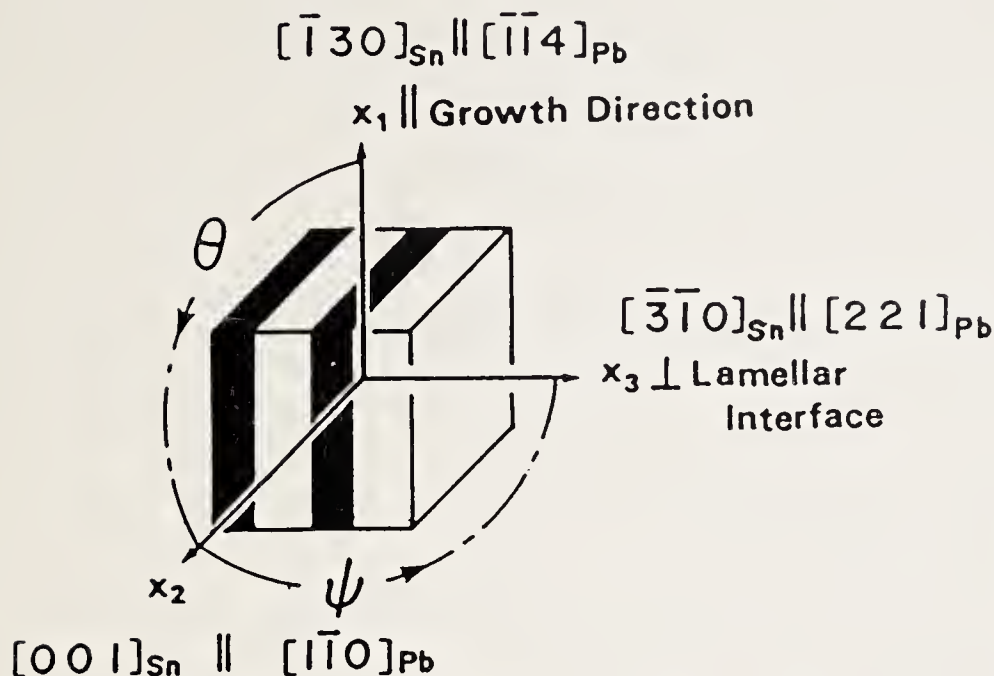


Figure 2. Crystallographic orientation relationships in the Pb-Sn lamellar eutectic.

Mason (1976) and Huntington (1958) may be converted to the coordinate system of our specimen in the specific crystallographic orientation depicted in figure 2. The matrix computation of the lamellar Pb-Sn eutectics in our orientation yields the elastic constants for Sn and Pb written as:

$$(C')_{\text{Sn}} = \begin{bmatrix} 7.76 & 2.49 & 2.58 & 0 & 0 & 0 \\ 2.49 & 8.76 & 2.49 & 0 & 0 & 0 \\ 2.58 & 2.49 & 7.76 & 0 & 0 & 0 \\ 0 & 0 & 0 & 2.20 & 0 & 0 \\ 0 & 0 & 0 & 0 & 2.45 & 0 \\ 0 & 0 & 0 & 0 & 0 & 2.20 \end{bmatrix},$$

$$(C')_{\text{Pb}} = \begin{bmatrix} 5.10 & 3.81 & 3.60 & 0 & 0 & 0 \\ 3.81 & 5.73 & 2.97 & 0 & 0 & 0 \\ 3.60 & 2.92 & 5.88 & 0 & 0 & 0 \\ 0 & 0 & 0 & 0.49 & 0 & 0 \\ 0 & 0 & 0 & 0 & 1.12 & 0 \\ 0 & 0 & 0 & 0 & 0 & 1.32 \end{bmatrix}.$$

Furthermore, the law of mixtures, for materials consisting of volume fractions, of matrix, V_I , and of fibre, V_{II} may be expressed to obtain elastic constants of multi-phase eutectics. The formulae for calculating them are given in a tabular form in table 2.

The elastic constants of the eutectic Sn-Pb alloy are calculated in accordance with the rule of mixtures as follows (Huntington 1958; Mason 1976),

$$(C')_{\text{eutectic}} = \begin{bmatrix} 6.99 & 2.98 & 2.93 & 0 & 0 & 0 \\ 2.98 & 7.66 & 2.65 & 0 & 0 & 0 \\ 2.93 & 2.65 & 6.97 & 0 & 0 & 0 \\ 0 & 0 & 0 & 0.96 & 0 & 0 \\ 0 & 0 & 0 & 0 & 1.70 & 0 \\ 0 & 0 & 0 & 0 & 0 & 1.87 \end{bmatrix}.$$

The experimental results obtained in this investigation through the measurements of

Table 2. Rule of mixtures for elastic constants of eutectic composite.

$C_{11} = V_I C_{11}^I + V_{II} C_{11}^{II} + \frac{(C_{13}^{II} - C_{13}^I) \cdot V_I \cdot V_{II} \cdot C_{13}^I}{V_{II} C_{33}^I + V_I C_{33}^{II}} - \frac{(C_{13}^{II} - C_{13}^I) \cdot V_{II}^2 \cdot C_{13}^{II}}{V_I C_{33}^I + V_{II} C_{33}^{II}}$	
$C_{22} = V_I C_{22}^I + V_{II} C_{22}^{II} + \frac{(C_{23}^{II} - C_{23}^I) \cdot V_I \cdot V_{II} \cdot C_{23}^I}{V_{II} C_{33}^I + V_I C_{33}^{II}} - \frac{(C_{23}^{II} - C_{23}^I) \cdot V_{II}^2 \cdot C_{23}^{II}}{V_I C_{33}^I + V_{II} C_{33}^{II}}$	
$1/C_{33} = \frac{V_I}{C_{33}^I} + \frac{V_{II}}{C_{33}^{II}}$	$C_{66} = V_I C_{66}^I + V_{II} C_{66}^{II}$
$C_{12} = V_I C_{12}^I + V_{II} C_{12}^{II}$	
$1/C_{44} = \frac{V_I}{C_{44}^I} + \frac{V_{II}}{C_{44}^{II}}$	$1/C_{13} = \frac{V_I}{C_{13}^I} + \frac{V_{II}}{C_{13}^{II}}$
$1/C_{55} = \frac{V_I}{C_{55}^I} + \frac{V_{II}}{C_{55}^{II}}$	$1/C_{23} = \frac{V_I}{C_{23}^I} + \frac{V_{II}}{C_{23}^{II}}$

the velocity of the elastic waves in the eutectic solid are given below,

$$(C')_{\text{eutectic}} = \begin{bmatrix} 7.12 & 4.06 & 3.85 & 0 & 0 & 0 \\ 4.06 & 7.29 & 3.85 & 0 & 0 & 0 \\ 3.85 & 3.85 & 6.62 & 0 & 0 & 0 \\ 0 & 0 & 0 & 1.38 & 0 & 0 \\ 0 & 0 & 0 & 0 & 1.42 & 0 \\ 0 & 0 & 0 & 0 & 0 & 1.53 \end{bmatrix}$$

Here, the elastic constants for the mixture have been assumed to be orthotropic. A comparison of earlier and present results reveals that the elastic constants along the diagonal are quite close to each other but the off-diagonal elements in the present investigation are slightly greater. The elastic constants obtained by us and those obtained by Verhoeven *et al* (1977) are illustrated in figure 3, from which it is apparent that these two give a reasonable agreement and that the elastic constants of this material is anisotropic to a considerable degree, the reason for this being that the eutectic specimens obtained in this experiment had crystallographically oriented directional microstructures. From the measured values of elastic constants were computed the elastic constants in different directions as rotated around the axis of the normal interface, one of the examples being shown in figure 3.

3.4 Electrical resistivity of the composite

The result of resistivity measurements can be described as follows:

$$\rho = \begin{bmatrix} 9.11 & 0 & 0 \\ 0 & 9.06 & 0 \\ 0 & 0 & 27.35 \end{bmatrix},$$

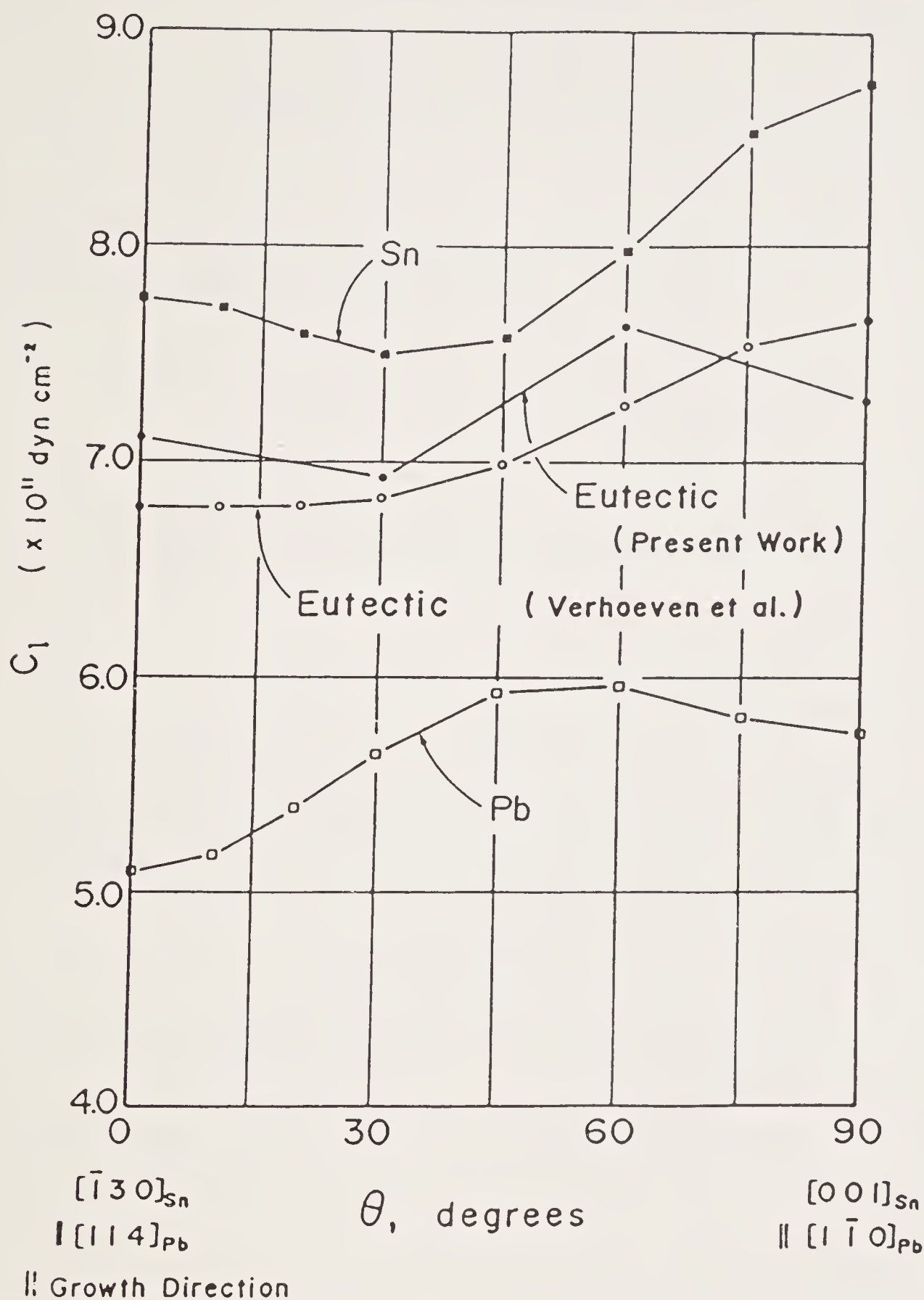


Figure 3. Orientation (θ) dependence of longitudinal modulus (C_l) of Pb-Sn single eutectic crystal.

in units of $\mu\Omega \text{ cm}$ for Pb-Sn eutectics, and $\rho = 13.09$ and $8.03 \mu\Omega \text{ cm}$ for specimens grown at 12 and 30 mm/hr, being measured in parallel with the growth direction. When these values are compared with those of previous investigators after transforming their results to our crystallographic orientation, our results give much greater resistivity in the direction of x , but those in the other directions are, on the other hand, smaller.

This discrepancy is believed to arise from an influence of the interfacial straining field. The difference in plate-like phases in contact can result from the degree of adhesion. The degree of coherency at the two-phase boundary might have influenced the resistivity across the interface.

4. Conclusions

The following conclusions are drawn from this investigation:

(1) With the coordinate system as shown in figure 2, the elastic constants were determined to be

$$(C')_{\text{eutectic}} = \begin{bmatrix} 7.12 & 4.06 & 3.85 & 0 & 0 & 0 \\ 4.06 & 7.29 & 3.85 & 0 & 0 & 0 \\ 3.85 & 3.85 & 6.62 & 0 & 0 & 0 \\ 0 & 0 & 0 & 1.38 & 0 & 0 \\ 0 & 0 & 0 & 0 & 1.42 & 0 \\ 0 & 0 & 0 & 0 & 0 & 1.53 \end{bmatrix}.$$

(2) The resistivity along the different crystallographic directions is anisotropic, given by

$$(\rho_{\text{eutectic}}) = \begin{bmatrix} 9.11 & 0 & 0 \\ 0 & 9.06 & 0 \\ 0 & 0 & 26.37 \end{bmatrix}.$$

(3) The anisotropic nature of Pb-Sn eutectics is considerable, as computed for the specimen with different rotational angles along the axis normal to the interface of the lamellae. A typical example has been shown.

The authors express their sincere thanks to Mita Industrial Co., Ltd. for financial support in this investigation.

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Microstructural characterization of unidirectionally frozen *in situ* Sn-Se eutectic composites

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Abstract. An array of lamellar SnSe-SnSe₂ structure is obtained by unidirectional solidification, in which SnSe and SnSe₂ are, respectively, *p* and *n* type semiconductors. Their structural morphology was examined with use of transmission and scanning electron microscopes. It was found that the ordering of the alternative layers of the phases could be accomplished by a suitable choice of freezing rates, although several kinds of structural defects such as terminations, misfit lamellae and colony structure were observed. The mechanisms of these defect formations were considered in terms of the constitutional supercooling. Furthermore, the crystallographic relationship between the two phases in the solidified state was determined.

Keywords. Sn-Se eutectic; composite semiconductor; constitutional supercooling; defect structure; crystallography.

1. Introduction

SnSe and SnSe₂ are, respectively, *p* and *n* type semiconducting compounds. The eutectic alloy of SnSe and SnSe₂ produces alternative layers of each phase and it may be considered a multi-layered *p-n* composite. The thickness of the layers can be controlled easily by varying solidification parameters. This enables the use of this alloy as a potential material having semiconducting properties. This investigation deals with the microstructural characterization and electrical properties of the eutectic SnSe-SnSe₂ composition.

2. Experimental method

2.1 Preparation of SnSe, SnSe₂ and their eutectic composite

The purity of Sn and Se elements used was 99.999%. Sn and Se are weighed to make Sn – 50.0 at.% Se for SnSe, Sn – 66.6 at.% Se for SnSe₂ and Sn – 61.0 at.% Se for eutectic

SnSe-SnSe₂ alloy. Each of the weighed elements was encapsulated in a quartz tube with an inner diameter of 8 mm which had been evacuated down to a pressure of approximately 0.01 Pa. Of them, SnSe₂ polycrystals and eutectic SnSe-SnSe₂ alloys were subsequently directionally solidified by the Bridgman technique under a temperature gradient of 1333 K/m.

Single crystals of SnSe were grown by the vapour transport technique in a closed tube (Yu *et al* 1981).

2.2 Microstructural examinations of the eutectic alloys

Eutectic alloys which had been directionally solidified were examined in a scanning electron microscope and a transmission electron microscope, which are respectively referred to as SEM and TEM hereafter. SEM observations were made both in the longitudinal and the lateral directions. In doing so, the specimens were cut out by a fine cutter, polished with emery paper, and finally cloth-polished in order to obtain a smooth surface. After successive polishings, it was etched for about 30 seconds in a solution consisting of one part of 50% HF and one part of 50% HNO₃ by volume (Yue & Yu 1982). In TEM observations, thin film specimens were peeled off from the cut surface of the eutectic mixture with the cleavage face parallel to the surface of the film.

2.3 Resistivity and Hall coefficient measurements of SnSe and SnSe₂

Resistivity and Hall coefficient were measured by Van der Pauw's method (1958). SnSe₂ polycrystalline specimens were cut into thin films 0.4 mm thick with faces parallel to the solidification direction and then polished to obtain a smooth surface. A SnSe wafer specimen of about the same thickness as the above was cut with the cleaved face parallel to (001) of the single crystal. The specimens were jointed with a platinum wire and pure In was evaporated onto them at 1.3×10^{-3} Pa pressure, while silver paste was used in order to establish Ohmic contact. On measuring both resistivities and Hall coefficients, a constant current of 5 mA was passed through. In the case of Hall coefficient measurements, the magnetic field of 0.522 wb/m² was applied. The temperature was varied from 153 K to 593 K.

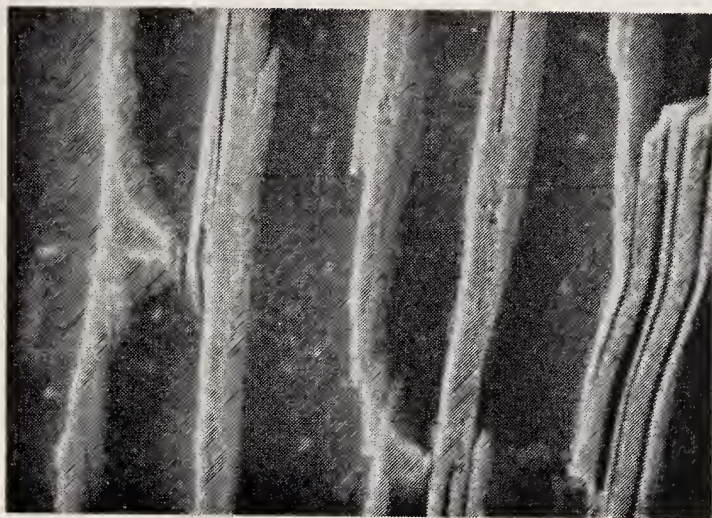
2.4 Preparation of single p-n junction

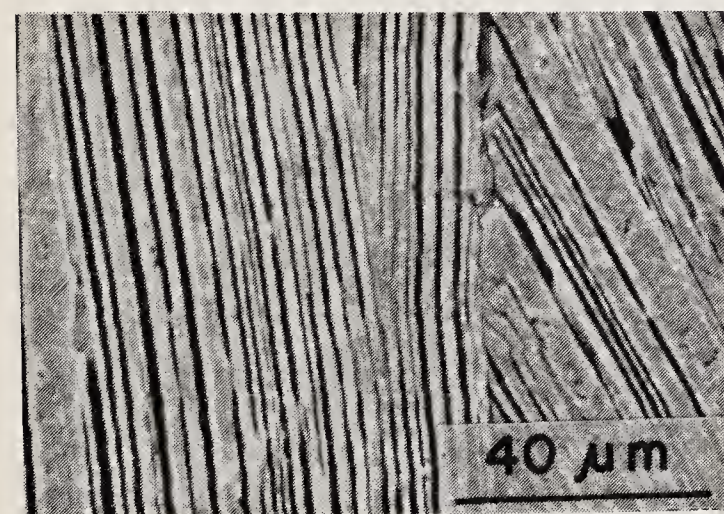
A single p-n junction was prepared by depositing SnSe₂ onto single crystalline SnSe within the reduced pressure of about 1.33×10^{-3} Pa, wherein the temperature of the substrate, SnSe₂, was kept at 393 K.

3. Experimental results

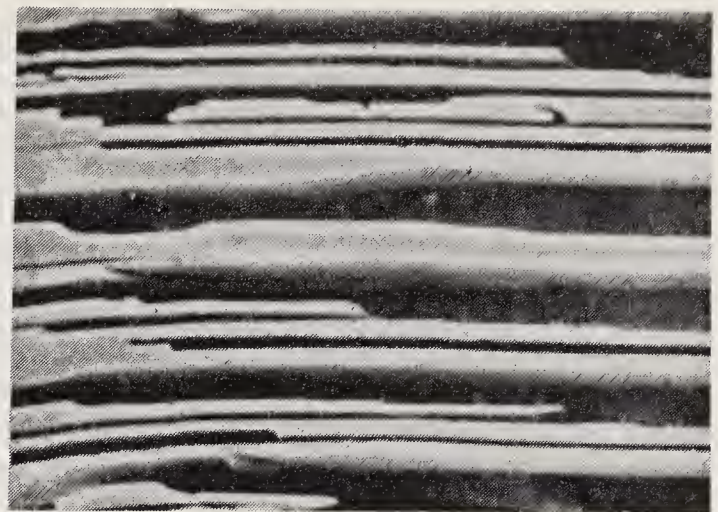
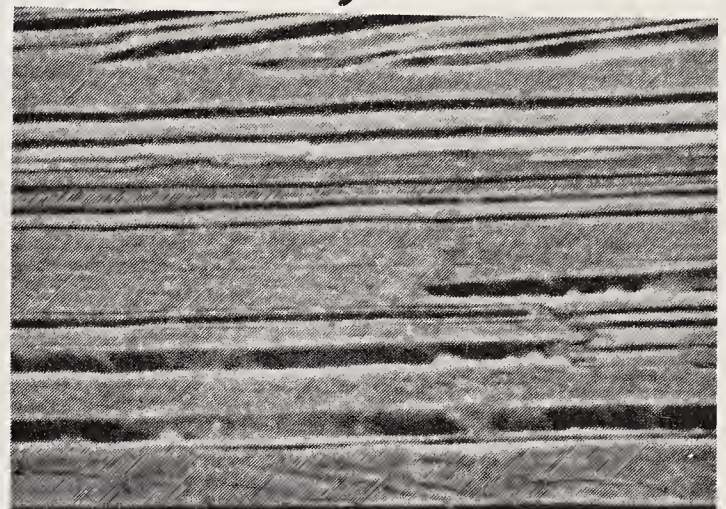
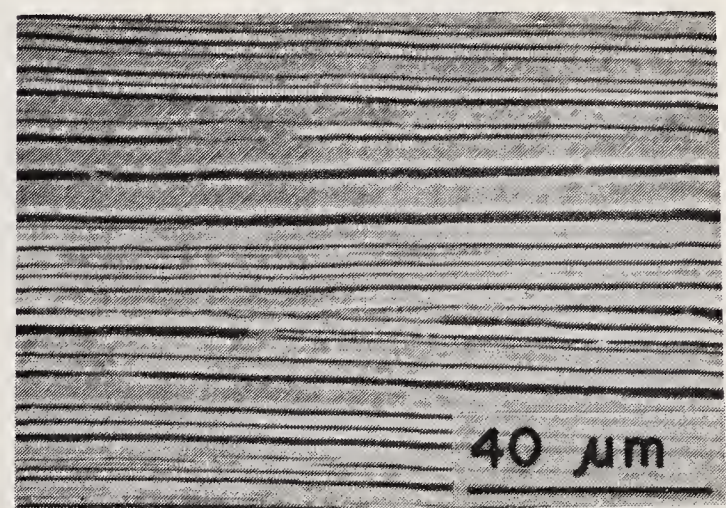
3.1 Microstructure of eutectic SnSe-SnSe₂ alloy

Figures 1 and 2 show the SEM micrographs of the specimens grown at varying rates of 0.277 to 142 μ m/s. It is clear that the greater the rate of solidification, the finer the interlamellar spacings, although the spacings of the lamellae are scattered in a considerable range of width. An array of the ordering and defect-free structure was favoured by the solidification rate of 4 μ m/s under these conditions. Rates which are lower or greater than this value could cause an increase in the deterioration of the morphology of the layered structure. Identification of SnSe and SnSe₂ phases was made


 $v = 0.277 \mu\text{m} / \text{s}$

 $v = 1.11 \mu\text{m} / \text{s}$

 $v = 2.22 \mu\text{m} / \text{s}$

Transverse section


 $v = 0.277 \mu\text{m} / \text{s}$

 $v = 1.11 \mu\text{m} / \text{s}$

 $v = 2.22 \mu\text{m} / \text{s}$

Longitudinal section

Figure 1. Scanning electron micrograph of SnSe-SnSe₂ eutectic at different growth velocities.

by the electron probe microanalyser, the results of which are illustrated in figure 3. In the figure, the phase designated as A corresponds to the phase rich in Sn and deficient in Se. Thus, the A-phase is identified as SnSe and similarly the B-phase as SnSe₂. Figure 4 illustrates the TEM micrograph of a film cleaved off from SnSe single crystals. The crystal structure of SnSe is orthorhombic with lattice parameters of $a = 0.446$, $b = 0.419$ and $c = 1.57$ nm. Figure 4 is the picture taken with the zone axis along the c -axis, i.e., $[001]$ and from this it is clear that the cleavage planes are those containing the zone axes of $\langle 010 \rangle$ and $\langle 110 \rangle$.

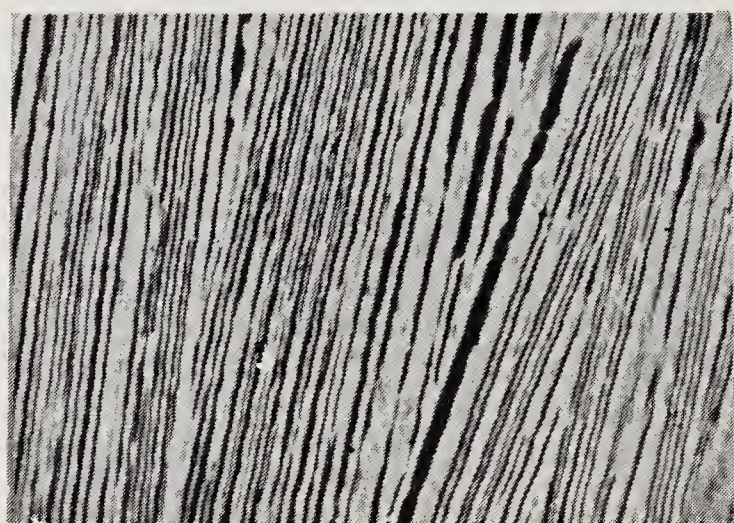
Figure 5 shows the TEM picture of SnSe₂ single crystals having the hexagonal unit cell



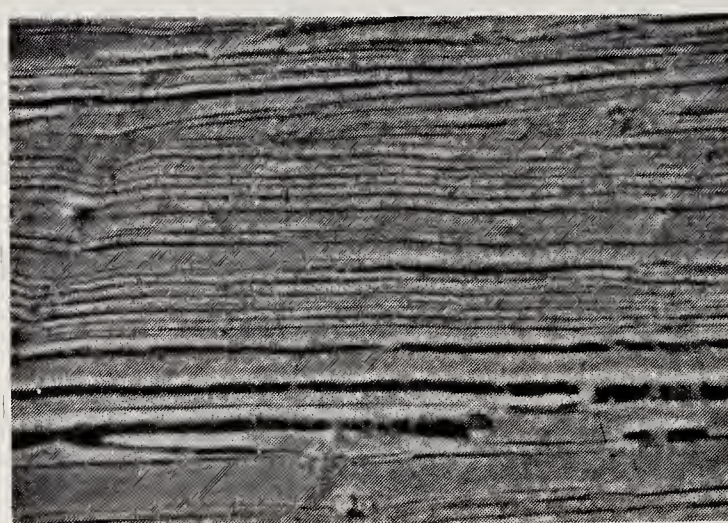
$v = 4.44 \mu\text{m} / \text{s}$



$v = 4.44 \mu\text{m} / \text{s}$



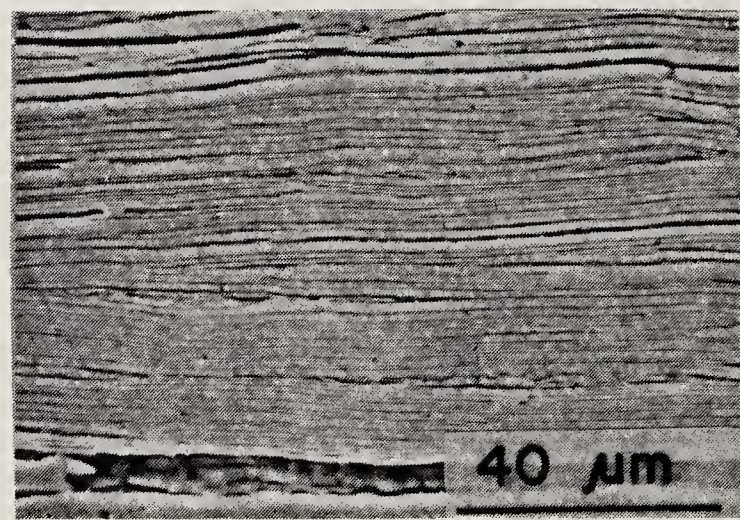
$v = 17.7 \mu\text{m} / \text{s}$



$v = 17.7 \mu\text{m} / \text{s}$



$v = 142 \mu\text{m} / \text{s}$



$v = 142 \mu\text{m} / \text{s}$

Transverse section

Longitudinal section

Figure 2. Scanning electron micrograph of SnSe-SnSe₂ eutectic at different growth velocities.

with $a = 0.38$, $c = 0.61$ nm. The beam passes through the SnSe₂ crystal along the c -axis, showing that the cleavage planes are, one that contains the a -axis and the other that is normal to the c -axis. Furthermore, innumerable moiré fringes can be observed in this structure.

Figure 6 indicates the TEM structure of the longitudinal section of the specimen growth at the rate of $4.44 \mu\text{m/s}$. Obviously the following crystallographic orientations

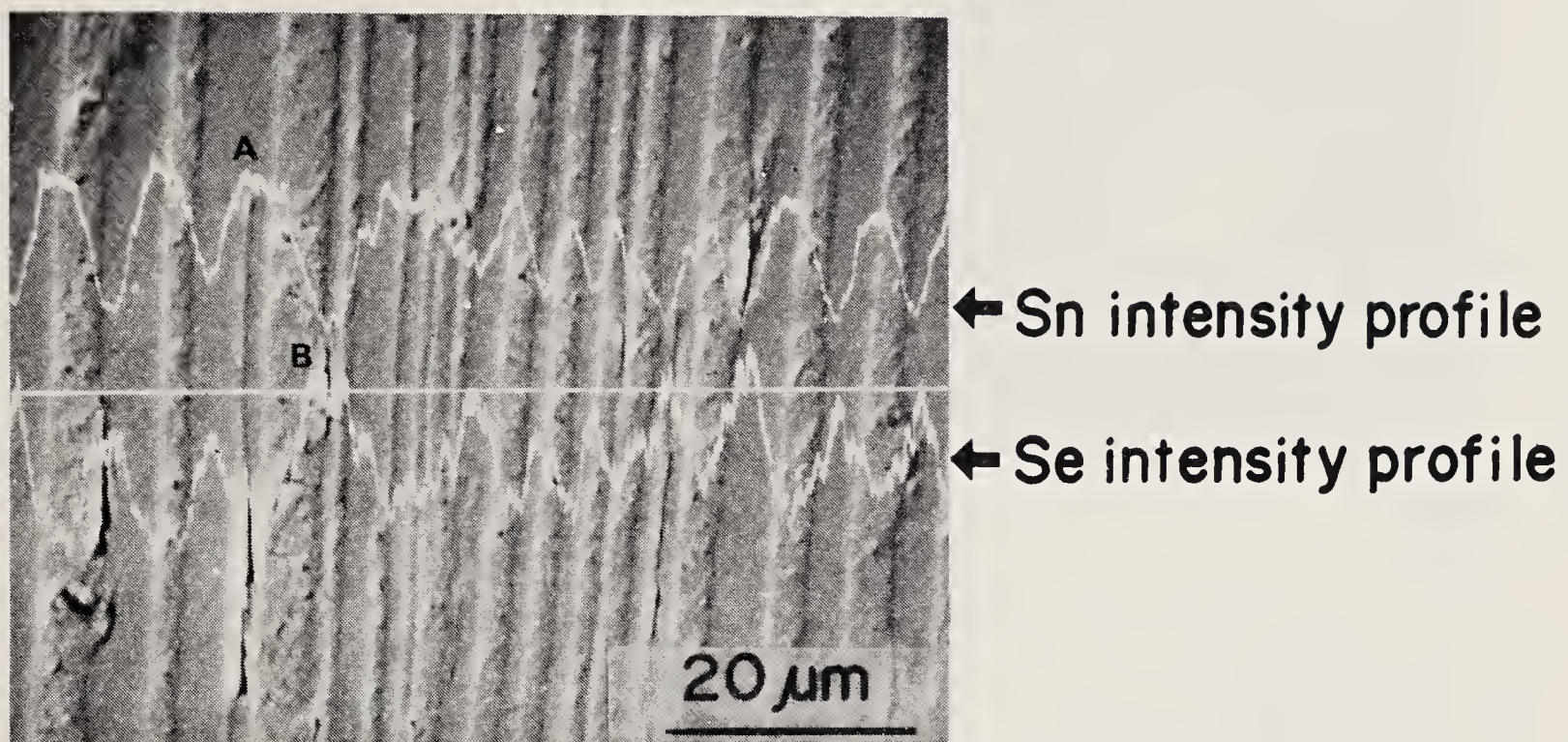
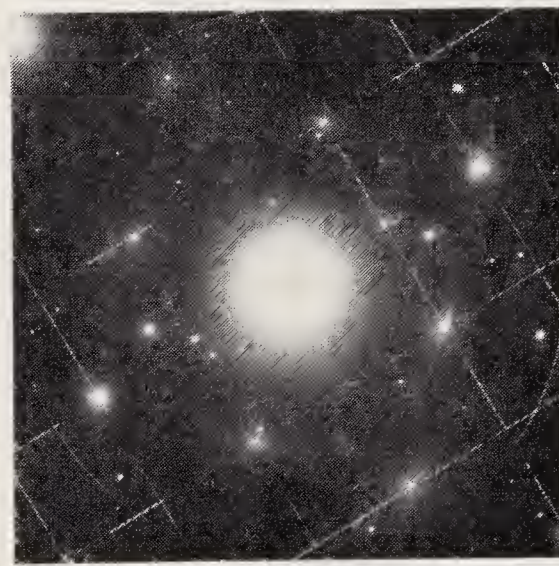
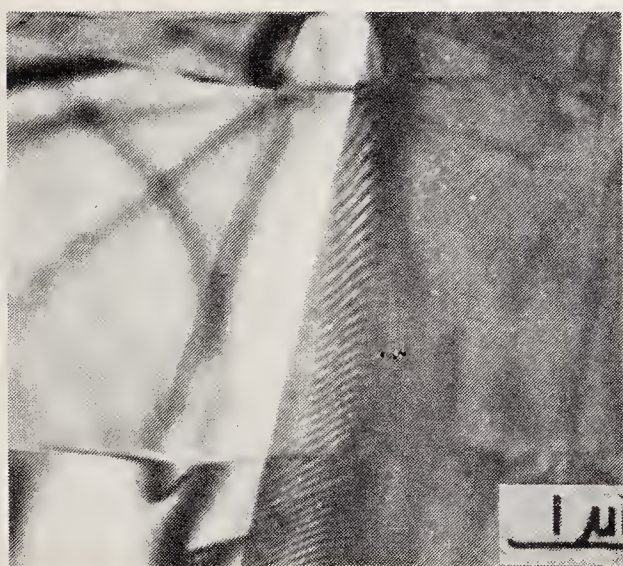
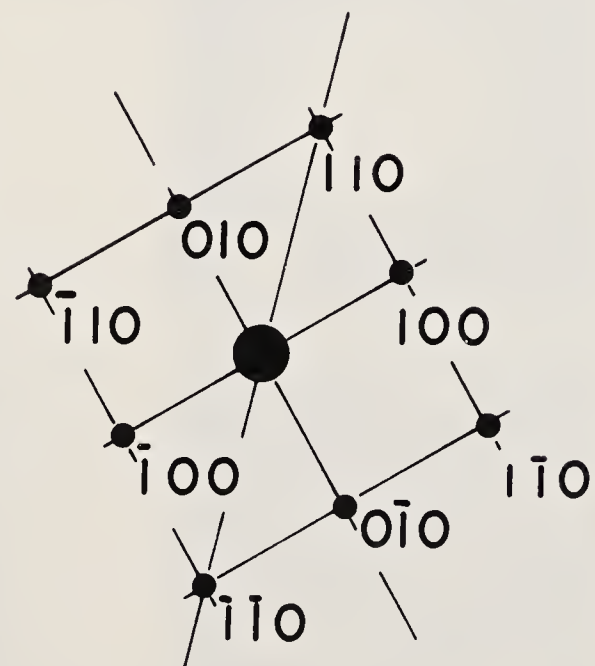
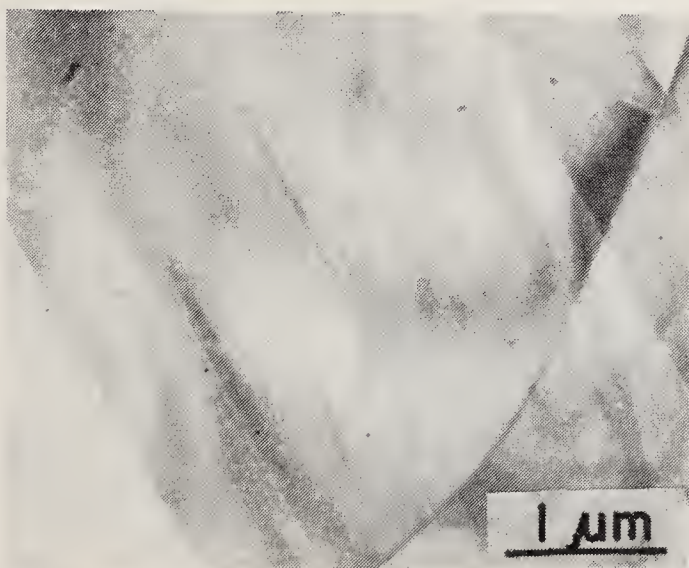


Figure 3. Scanning electron micrograph and linear analysis in the unidirectionally grown SnSe-SnSe₂ eutectic.

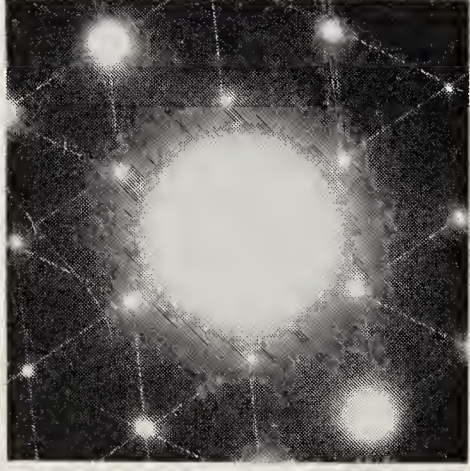
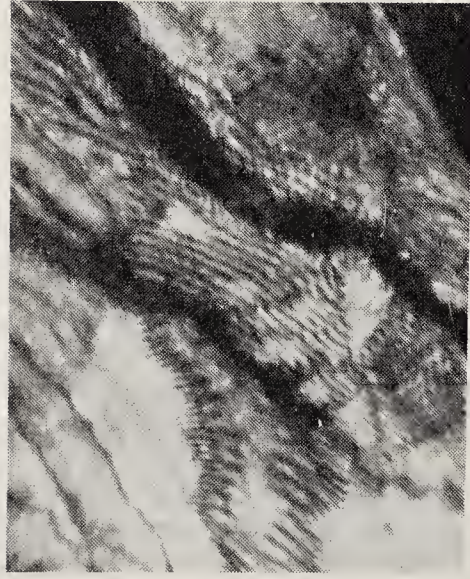


Cleaved SnSe phase in SnSe-SnSe₂ eutectic



Cleaved surface of SnSe single crystal with surface steps

Figure 4. Transmission electron micrograph of SnSe phase.



slightly rotate

Cleaved SnSe_2 phase in $\text{SnSe}-\text{SnSe}_2$ eutectics

Figure 5. Transition electron micrograph of SnSe_2 phase in $\text{SnSe}-\text{SnSe}_2$ eutectic.

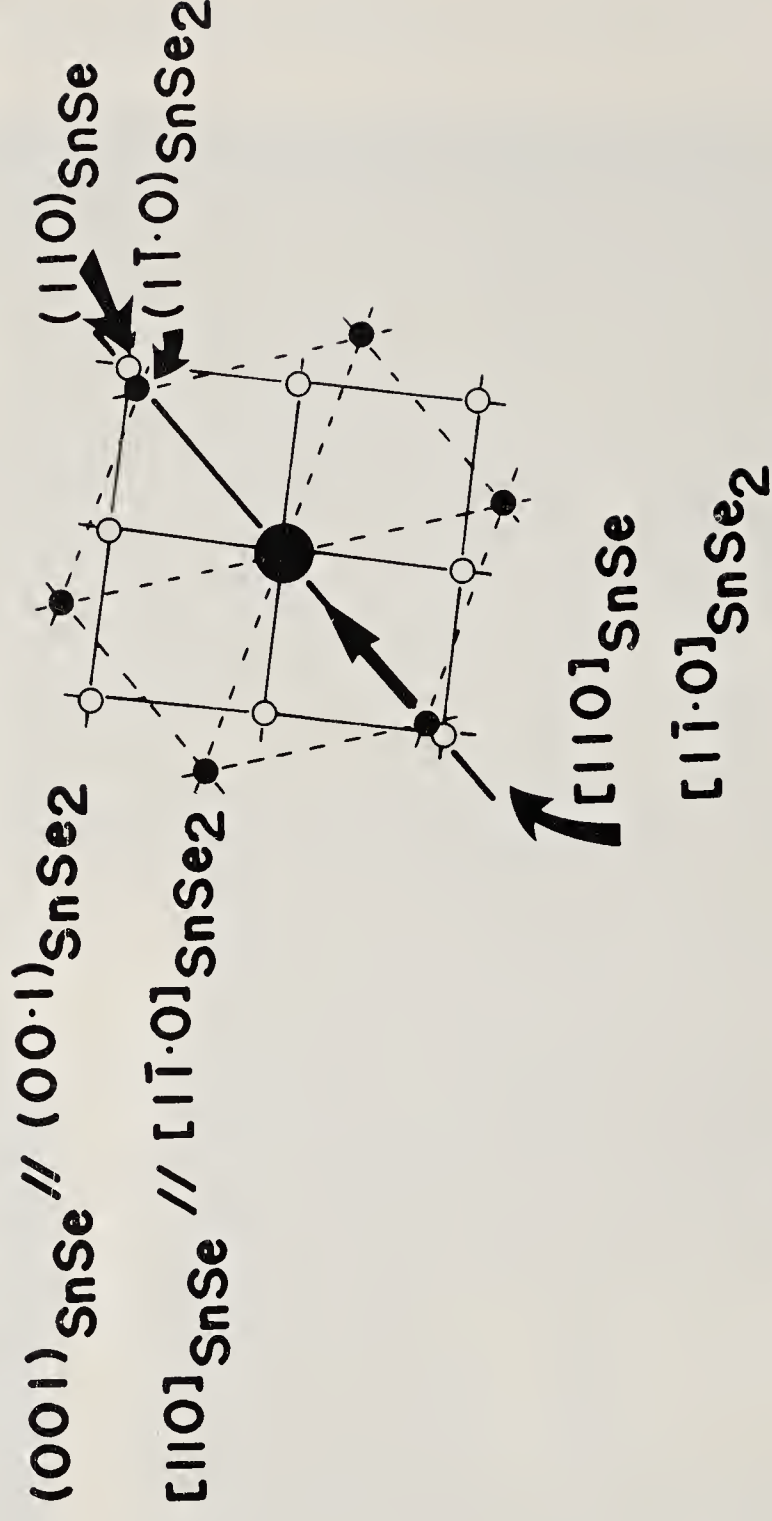
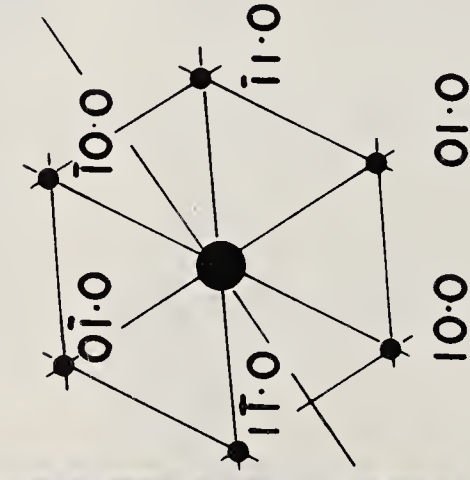
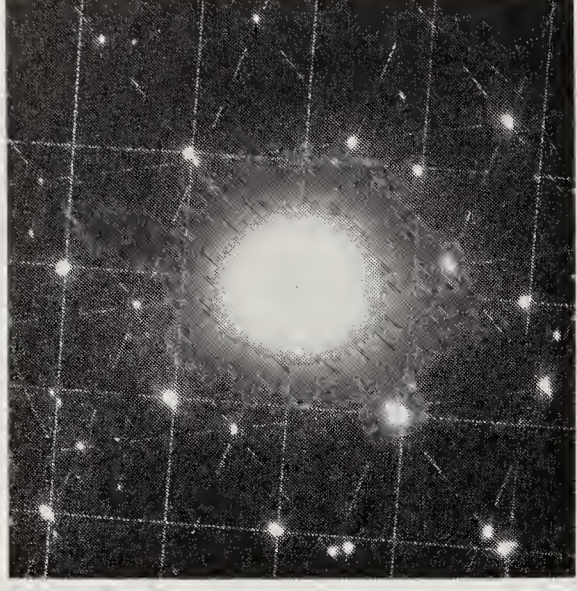
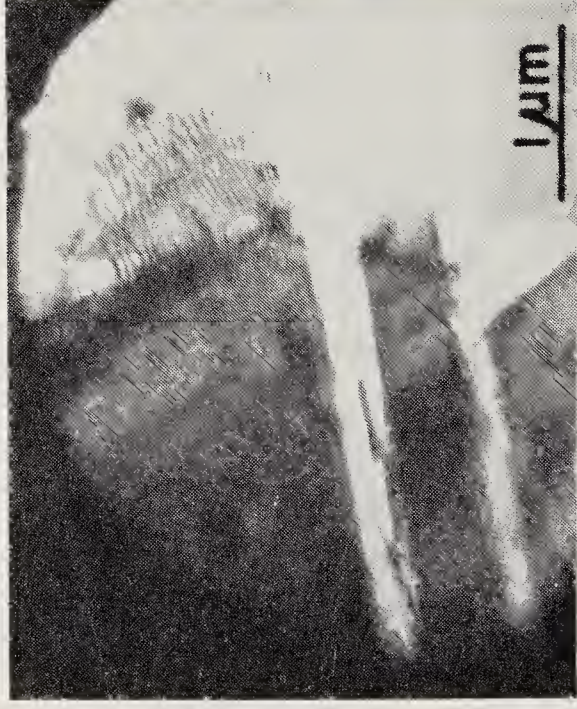


Figure 6. Transmission electron micrograph of $\text{SnSe}-\text{SnSe}_2$ eutectic.

are seen to exist:

$$\begin{aligned} &(001)_{\text{SnSe}} // (00\cdot1)_{\text{SnSe}_2} \\ &[110]_{\text{SnSe}} // [1\bar{1}\cdot0]_{\text{SnSe}_2}. \end{aligned}$$

In figure 7 is plotted the average lamellar spacings of SnSe and SnSe₂ vs. the unidirectional solidification rate on the logarithmic scale, from which the slope of the linear relationship is found to be 2.15. This suggests that the following Jackson-Hunt's relationship (Jackson & Hunt 1966) is approximately satisfied

$$\lambda^2 \cdot v = 2.8 \quad (\mu\text{m/s})^3/\text{s}. \quad (1)$$

3.2 Electrical properties of SnSe-SnSe₂

Figure 8 shows the temperature dependence of the resistivities of specimens which had been allowed to solidify at different rates. It is clear that in all the specimens at elevated temperatures, the resistivity decreases with increasing temperature and that at low temperatures it increases with decreasing temperature for SnSe₂ and eutectic SnSe-SnSe₂, whereas it decreases slightly with decreasing temperatures for SnSe.

Here, from the linear portion of figure 8 at an elevated temperature, the energy gap between the conduction band and the filled band is estimated to be 0.88 eV for SnSe single crystals, 1.0 eV both for SnSe₂ polycrystals and SnSe-SnSe₂ eutectic composites. Figure 9 demonstrates the temperature-dependence of the Hall coefficients measured from the same specimen shown in figure 8. The Hall coefficient of SnSe single crystals is essentially temperature-independent, whereas those of SnSe₂ polycrystals and SnSe-SnSe₂ eutectic mixtures fall rapidly with increasing temperature. Figure 10 shows the variation of mobilities at various temperatures. In all the specimens, the slope of the mobilities with respect to temperature are approximately the same. Figure 11 illustrates the current-voltage characteristic of a single *p-n* junction under illuminated or unilluminated conditions. It may be pointed out that the open circuit voltage of the specimens is 20 mV and the short circuit current is 0.4 mA.

4. Discussions

4.1 Directionally solidified SnSe-SnSe₂ eutectics

In order for SnSe-SnSe₂ eutectics to be used as potential material for semiconducting devices, it is required that each phase of SnSe and SnSe₂ be free of structural defects in the aspect of ordering and morphology. For this, occurrence of constitutional supercooling must be avoided. Generally speaking, the morphology of the liquid-solid interface is determined by the supercooling from the equilibrium solidification temperature:

$$\Delta T = \Delta T_C + \Delta T_\sigma. \quad (2)$$

The first term of (2) corresponds to the undercooling caused by the limited diffusivity at the planar front, which is given as:

$$\Delta T_C = m(C_E - C_x), \quad (3)$$

where *m* is the slope of the liquidus temperature in the phase diagram and *C_x* the

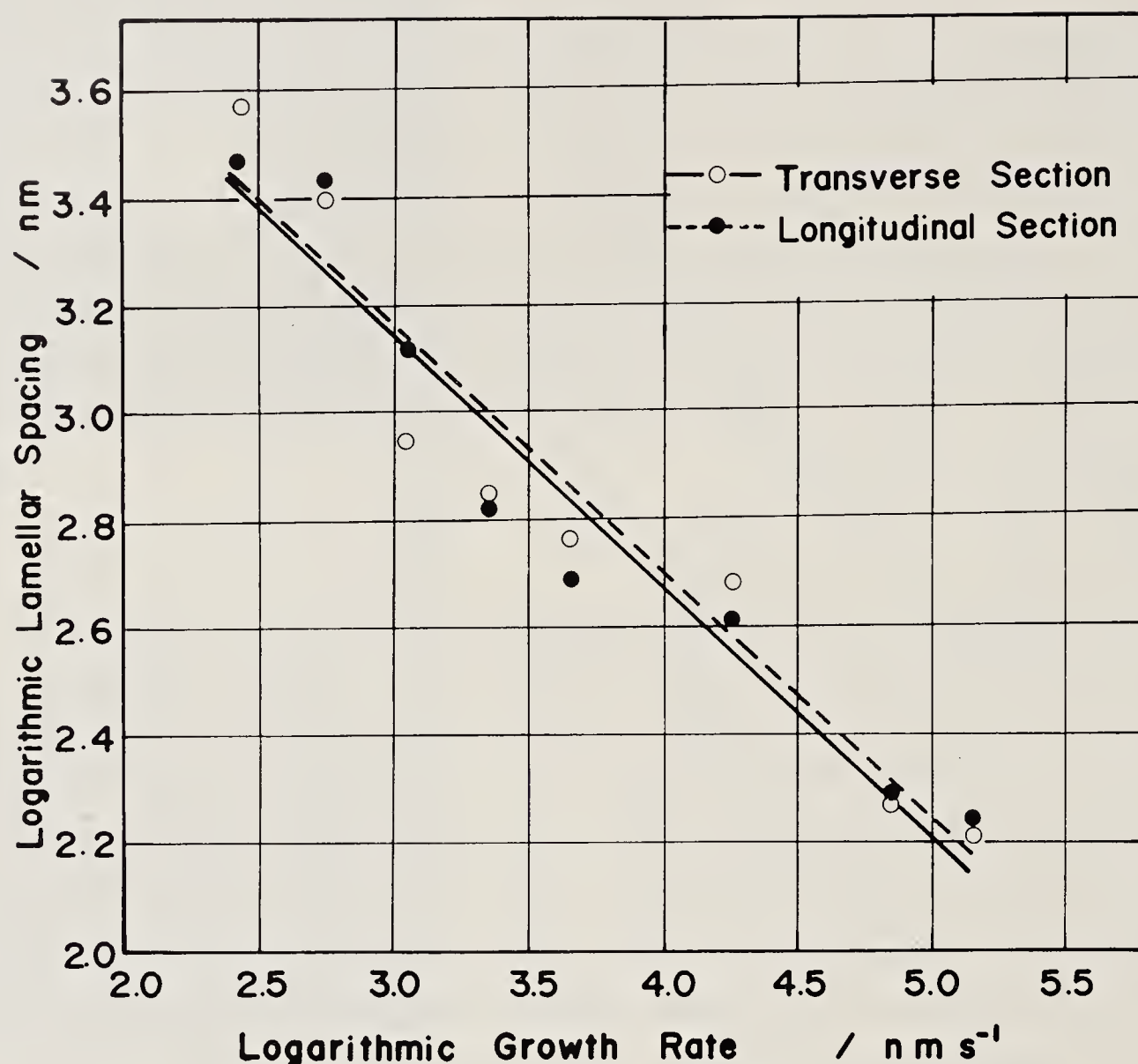


Figure 7. Logarithmic plot of lamellar spacing vs. growth rate for a SnSe-SnSe₂ eutectic.

composition at the solid-liquid interface. The second term on the right of (2) corresponds to the undercooling caused by the presence of the curvature of the front and it is given approximately by the Gibbs-Thomson equation (Ostwald 1901) as:

$$\Delta T_{\sigma} = aK_x = (T_E \sigma / L)(-d^2z/dx^2)[1 + (dz/dx)]^{-3/2}, \quad (4)$$

where a is given by $T_E \sigma / L$, T_E is the equilibrium temperature, σ the surface tension, L the latent heat, and K_x represents the radius of curvature of the front. Let us assume that both of these contributions are additive. Let SnSe and SnSe₂ phases be referred to as α and β -phases, respectively, for the sake of simplicity. The origin of the coordinate system is placed at the centre of α -phase on the solid-liquid boundary. Jackson & Hunt (1966) obtained the relationship under the condition that the solidification proceeds so as to minimize the total supercooling, ΔT . The relationship they obtained is:

$$\lambda^2 V = a^L / Q^L = \text{constant}, \quad (5)$$

where a^L and Q^L are, respectively, given by:

$$a^L = 2(1 + \zeta)[(a_{\alpha}^L / m_{\alpha}) + (a_{\beta}^L / \zeta m_{\beta})], \quad (6)$$

$$Q^L = p(1 + \zeta)^2 C_0 / (\zeta D), \quad (7)$$

where ζ is the relative thickness, S_{β} of the β layer divided by S_{α} of the α layer and S_{β} and S_{α} each are half the thicknesses of its own phase. P is a parameter associated with ζ , D the diffusivity, C_0 the width of the miscibility gap between α and β -phases,

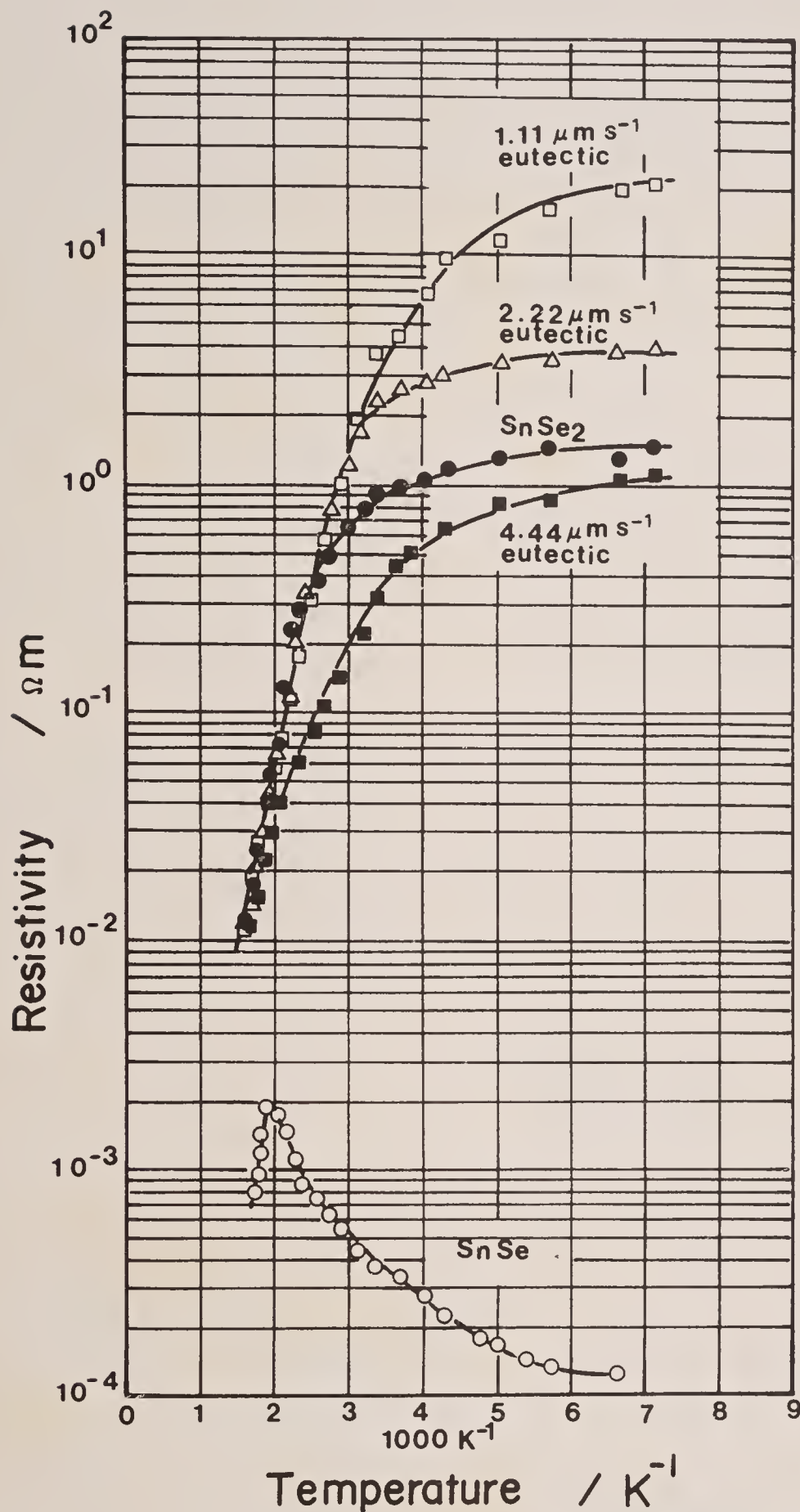


Figure 8. Temperature dependence of the resistivity for SnSe, SnSe₂ crystals and SnSe-SnSe₂ eutectic crystals.

m_α and m_β the slope of the liquidus line of each phase and a_α^L or a_β^L is the value of ΔT_σ of the α or β -phase.

From the experimental measurements for the SnSe-SnSe₂ eutectic alloy, it was found that $\lambda^2 V = a^L/Q^L = 2.8 (\mu\text{m})^3/\text{s}$. This is a little smaller than the values found elsewhere for most other eutectic alloys. From the phase diagram of figure 12, we see that $C_0 = 0.166$ atomic fraction, $m_\alpha = 2000 \text{ K}$ and $m_\beta = 303 \text{ K}$. All of these tend to reduce the value, a^L/Q^L , as tentatively compared with other eutectic alloy cases.

Adopting the previously given m_α , m_β and C_0 , with the rest of the other parameters being assumed to be the same, rough estimate demands that in order for the value, a^L/Q^L , to be small, the value a^L should be small, leading to a small value of σ/L .

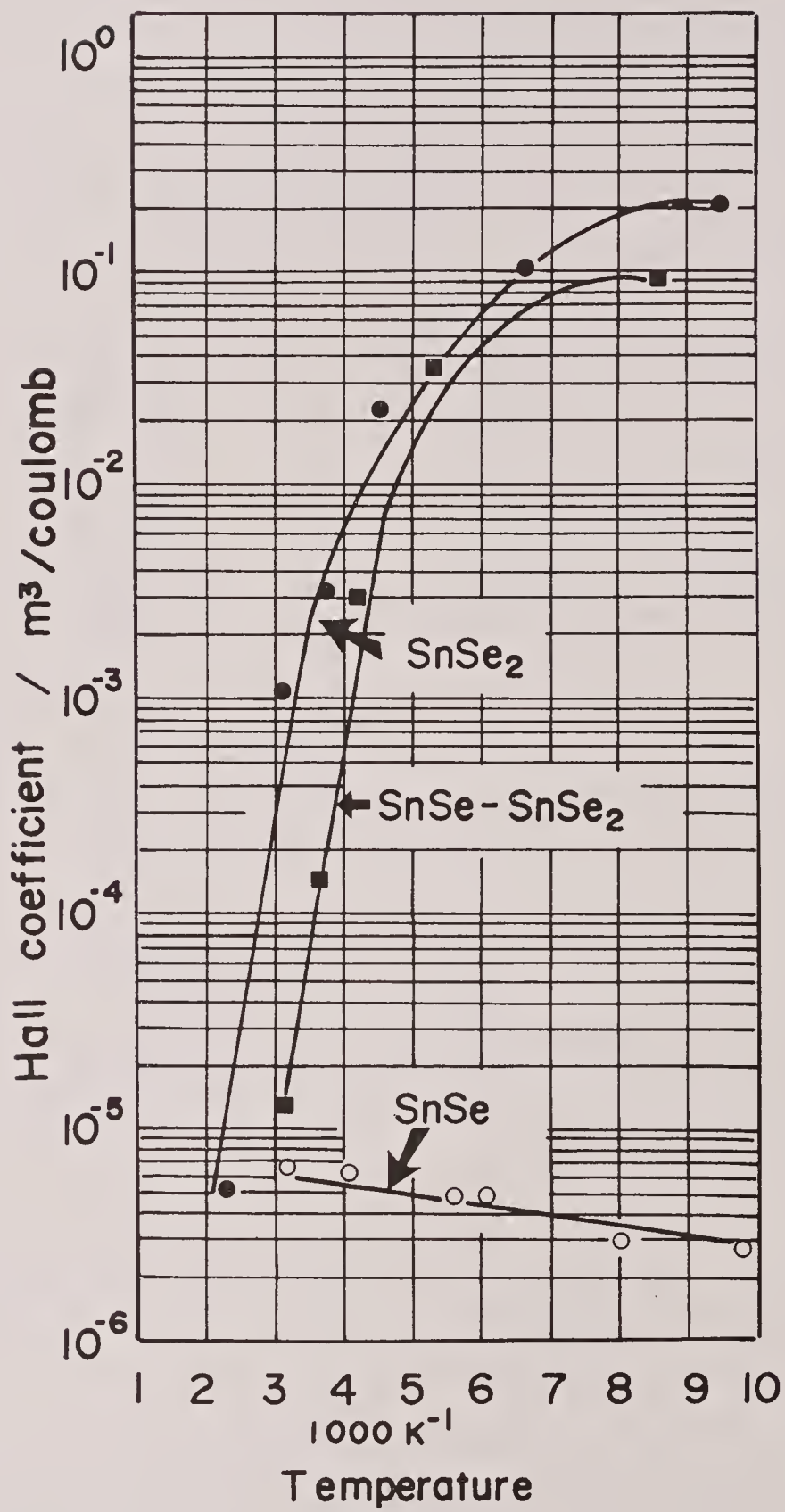


Figure 9. Temperature dependence of Hall coefficients of SnSe, SnSe₂ and SnSe-SnSe₂ eutectics.

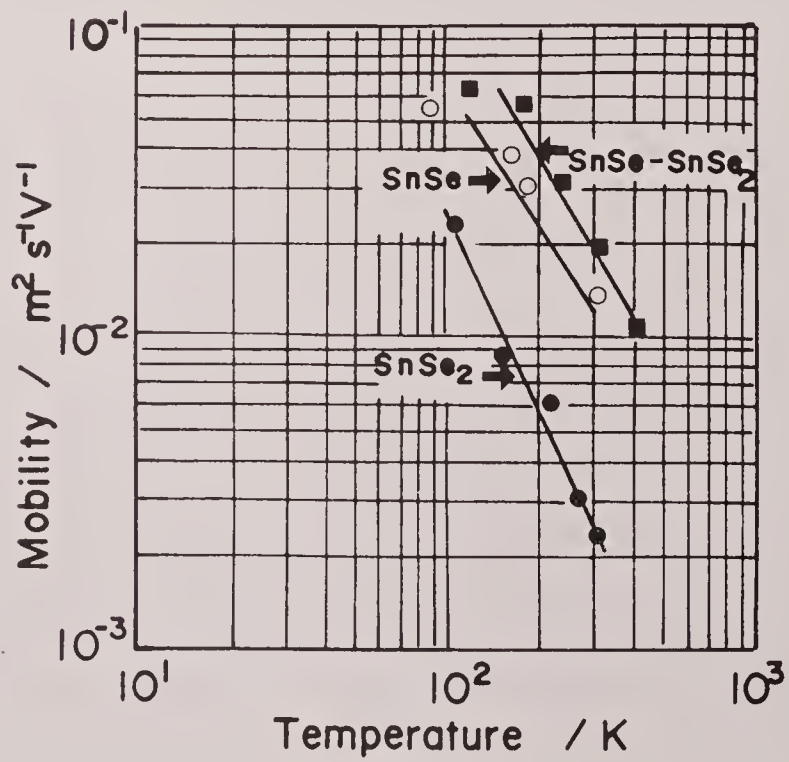


Figure 10. Temperature dependence of Hall mobilities in SnSe, SnSe₂ and SnSe-SnSe₂ eutectics.

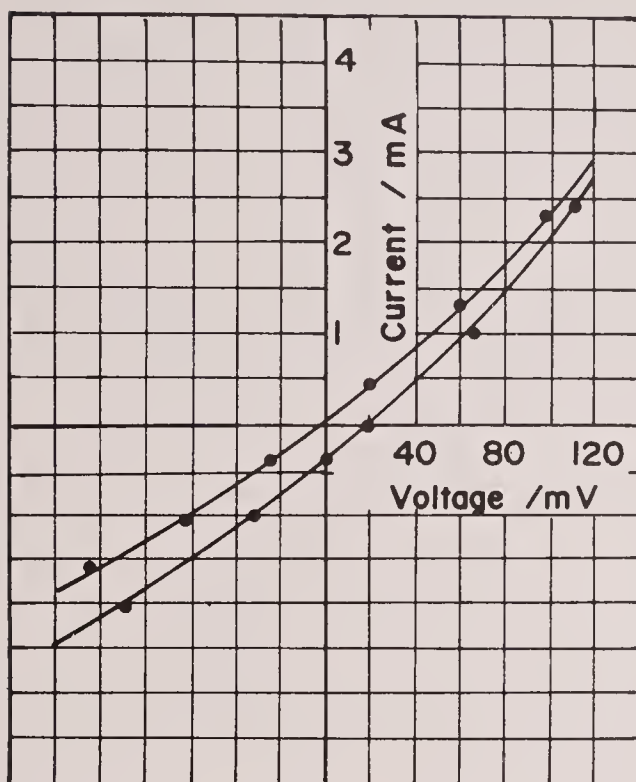


Figure 11. Illustration of the current-voltage characteristics of a single p - n junction under illuminated or unilluminated (the line passing through the origin) conditions.

Considering the interfacial energy between the α -phase and the liquid to be approximately of the same order of magnitude as that between the β -phase and the liquid, the latent heat of the β -phase must be great.

Let us now consider the morphology of the boundary at the solidification front with the minimum undercooling principle. Jackson & Hunt (1966) derived the following expression of the curved front of the α -phase i.e., in the region, $0 < x < S_\alpha$:

$$\frac{dz}{dx} = \tan \sin^{-1} \left\{ -\sin \theta \left[\frac{x}{\lambda} 2(1 + \zeta) + (\zeta + \eta) \left(\frac{2x}{\lambda} - \frac{2}{(1 + \zeta)P\lambda} \int_0^x f(x) dx \right) \right] \right\}, \quad (8)$$

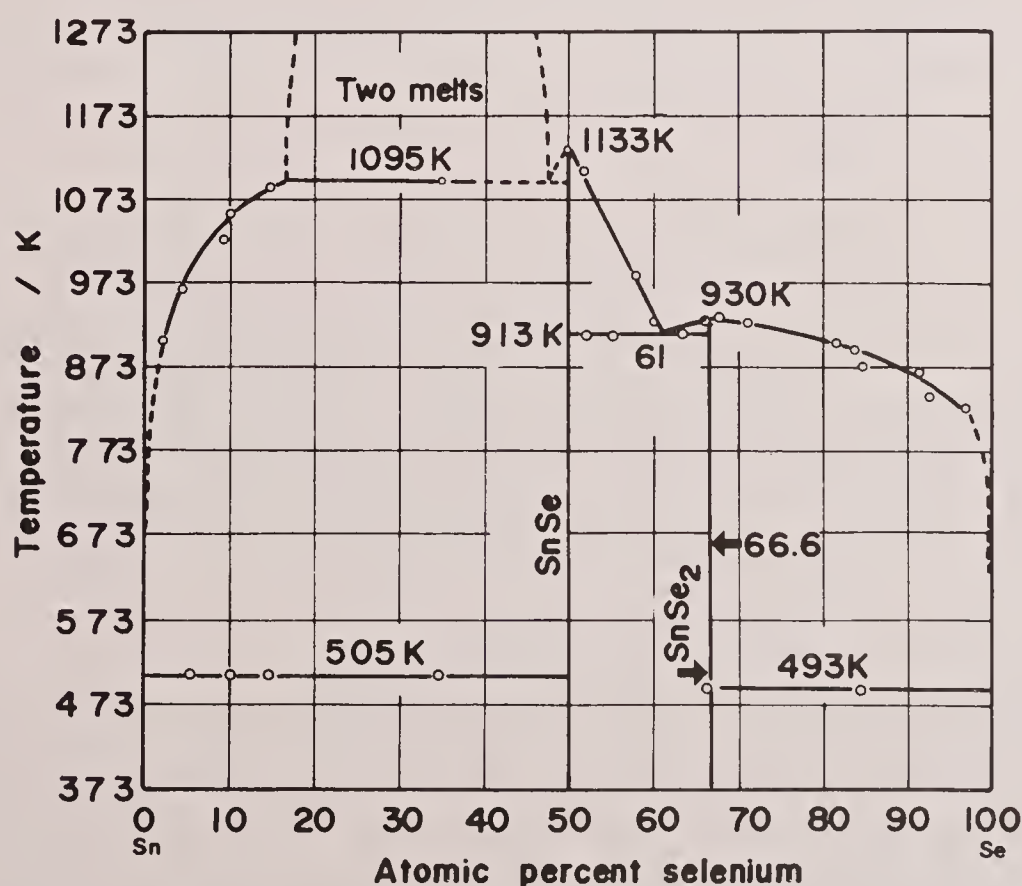


Figure 12. Equilibrium phase diagram of the Sn-Se binary system.

and the curved front of the β -phase, i.e., in the region, $S_\alpha < x < S_\alpha + S_\beta$:

$$\frac{dz}{dx} = \tan \sin^{-1} \left\{ \sin \theta \left[\left(\frac{\lambda - 2x}{\lambda} \right) \left(\frac{1 + \zeta}{\zeta} \right) + \left(\frac{1}{\zeta} + \frac{1}{\eta} \right) \left(\frac{\lambda - 2x}{\lambda} - \frac{2\zeta}{(1 + \zeta)P\lambda} \int_0^x f(x) dx \right) \right] \right\}, \quad (9)$$

where

$$f(x) = \sum_{n=1}^{\infty} \frac{\sin [n\pi/(1 + \zeta)] \cos (2n\pi x/\lambda)}{(n\pi)^2},$$

$$\eta = a_\beta m_\beta / a_\alpha m_\alpha.$$

Although a_β and a_α are unknown in the case of SnSe-SnSe₂ eutectics, it can be tacitly assumed that since a_α or a_β is given by $T_E \sigma / L$, σ and L for SnSe and SnSe₂ are of the same order of magnitude, and thus, $a_\alpha = a_\beta$, then $\eta = 6.6$. Furthermore, $\zeta = 1.964$, $P = 0.025$ are obtained by computation (Jackson & Hunt 1966). When these values are inserted into (8) and (9), one obtains the expression for dz/dx along the solid-liquid interface. It is now apparent that the curvature of the solid-liquid boundary in front of the α -phase is convex toward the liquid whereas that in front of the β -phase is concave toward the liquid. This difference has arisen possibly because the value of η is excessively large, that is, the difference in the slopes m_α and m_β is grossly different, giving rise to a big value of η under the feasible assumption of $a_\alpha = a_\beta$.

The supercooling required for the growth condition used here can be computed as follows:

$$\Delta T = 2ma^L/\lambda$$

where

$$\frac{1}{m} = \frac{1}{m_\alpha} + \frac{1}{m_\beta}, \quad a^L = 2(1 + \zeta) \left(\frac{a_\alpha^L}{m_\alpha} + \frac{a_\beta^L}{\zeta m_\beta} \right)$$

and, here, $m = 263$ K. Adopting the suitable value used for this growth experiment, the range of the undercooling for our growth conditions,

$$\Delta T = 0.016 - 0.27 \text{ K},$$

is obtained, from which it is clear that the supercooling at the solid-liquid interface may be seen to be small. Under growth conditions which are substantially close to the equilibrium, rupture of the regular lamellae takes place more often when the difference in the slopes of the liquidus lines are large than when the driving force for the solidification is small. The optimum condition of the unidirectional growth rate was observed at $4.44 \mu\text{m/s}$ in this investigation for SnSe-SnSe₂ eutectics.

4.2 Electrical properties of SnSe-SnSe₂ eutectics

Numata (1950) investigated the principal carrier of electrical conduction in SnSe_x alloy with varying x . He found that when x is greater than 1.5, the carrier is an electron while x is less than 1.0, it is a positive hole. That is, when Sn is in excess, the alloy is a p -type semiconductor, and when Se is in excess, it is n -type. This indicates that SnSe-SnSe₂

eutectics having a regularly ordered lamellar structure have alternatively stacked multiple p - n junctions.

Figure 8 shows the variation of electrical resistivities as a function of the reciprocal of absolute temperature, indicating that the resistivity of SnSe_2 polycrystals varies in a manner similar to that of SnSe-SnSe₂ eutectics. Thus, it may be said that the resistivity of the eutectic is merely a contribution of the resistivity of a single phase of SnSe_2 . The same figure also shows that the resistivity change is influenced remarkably by the disorder of the lamellae at low temperatures, figure 9 demonstrates that the Hall mobility decreases with increasing temperature and it is seen from figure 10 that the mobility varies linearly with T^{-2} . This result agrees with the observations made by Asanabe (1959). This result also indicates that the mobility is mainly determined by the contribution from lattice vibrations, leading to the conclusion that the resistivity variation of SnSe_2 polycrystals and SnSe-SnSe₂ eutectic samples depends to a great extent upon the conduction electrons, that is, the decrease of the Hall coefficient results from the increase in conduction electrons due to the increase in number of ionized atoms. The drop of the Hall coefficients at elevated temperatures corresponds to the intrinsic conduction region. The Hall coefficients of SnSe single crystals, on the other hand, show a slightly positive temperature dependence. This is considered to be due to the dissolved impurity atoms. As Asanabe (1959) pointed out, the variation of the electrical resistivity of SnSe-SnSe₂ eutectics except in the intrinsic region is considered to be principally due to the single phase SnSe_2 , and, therefore, it may be said that the attendant property arises from the microstructural defects formed during the unidirectional freezing. Figure 4 proves that there is a considerable compositional fluctuation, since one can observe many Moiré fringes appearing in a single phase region. This may be another contributory factor for the increase in the electrical resistivities. Figure 11 shows the current-voltage characteristics for a single p - n junction made from SnSe and SnSe_2 , from which it turns out that the open circuit voltage is 20 mV and the short circuit current is 0.4 mA. These values are a little smaller than those found by Yue & Yu (1982). It may be said that the SnSe-SnSe₂ eutectic alloy is one of the potentially useful materials for solar cells if it is allowed to solidify without defects and compositional fluctuations.

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Unidirectionally cooled eutectic and eutectoidal composites of Al-Cu alloy

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Abstract. Al-Cu alloys having eutectic (Al-17.3 at.% Cu) or eutectoid (Cu-24 at.% Al) compositions were allowed to solidify unidirectionally. These two alloys were cooled from an elevated temperature in order to observe the microstructural characteristics. The freezing rate for obtaining a parallel and ordered structure is naturally much faster in eutectic than in eutectoid alloys although both the reaction temperatures are about the same (eutectic temperature = 548, eutectoid temperature = 565° C). The difference in the solidification rate is due to the diffusion rates of each atomic species in the liquid and the solid states. Similar defects were observed in both the specimens and their mechanisms of formation will be considered.

Keywords. Directional cooling; Al-Cu alloy; *in situ* composite; eutectic reaction; eutectoid reaction.

1. Introduction

In situ composites are made quite simply by using a phase-separable alloy system under controlled heat flow in order to obtain uniform distribution and a morphologically desired form of a hard phase embedded in a soft matrix phase. This technique can be applied to any crystal system, involving liquid to solid or solid to another solid, as long as the reaction products resulting from the transformation are separated. The morphology of reaction products can be altered but is limited by suitable choice of cooling parameters.

The morphological variation is governed by the mechanism of the reactions which take place during transformation. Since phase separation has to occur, the diffusion of atoms is unavoidable. Mathematical formulations of the growth laws on the basis of different models have been put forward to describe different mechanistic views. But it is a difficult task to check the validity of the models adopting different alloy systems because relative physical parameters such as diffusivities of atoms involved are altered in different alloy systems. Here, a critical comparison was attempted in a specific alloy system which has both eutectic and eutectoid reactions.

A suitable vehicle for this purpose was believed to be an Al-Cu alloy system which has both these reactions occurring at around the same temperature level but at different compositions.

2. Experimental method

2.1 Preparation of specimens with eutectic and eutectoidal compositions

Samples of Al and Cu, each 99.99% pure, were suitably weighed to obtain either eutectic (Al-17.3 at.% Cu) or eutectoid (Cu-24.0 at.% Al) alloys and encapsulated in quartz tubes within which the graphite crucible was placed in order to prevent the molten metal from reacting with the quartz tubes. The tube was evacuated down to a pressure of 10^{-4} Pa. At first, both mixtures were entirely and separately melted and solidified to obtain homogeneous ingots. Then the ingot of eutectic composition was allowed to solidify directionally while that of the eutectoidal composition was cooled in a similar manner. The temperature gradient for both alloys was 13.8 K/mm. The range of cooling rates used was quite different. For eutectics it ranged from 2.8 to 33.3 $\mu\text{m/s}$ whereas for eutectoids it ranged from 0.4 to 2.9 $\mu\text{m/s}$. The difference in the rate resulted from the conditions that produced a more or less regular array of plate-like phases, i.e. α -Al and θ (CuAl_2) for eutectics, and α -Cu and γ for eutectoids. After directional cooling of the specimens, their microstructures were examined by optical, scanning electron and transmission electron microscopy. Thin films for TEM observations were made by electro-polishing the sliced specimens in a solution consisting by volume of 5 parts of alcohol and 1 part of nitric acid. The temperature was kept at 233 K during thinning and the voltage was kept at about 10 V. The thin films thus obtained were examined by TEM of the type Hitachi Hu-125Ds.

3. Experimental results

3.1 Al-17.3 at.% Cu eutectic alloy

Figure 1 shows the phase diagram of the Al-Cu system having a typical eutectic transformation at 821 K. The liquid phase is transformed into solid α -Al and θ (CuAl_2) phases. Since the parent phase is liquid, the diffusion of atom species is expected to be much faster than in the solid state and so is the rate of solidification, giving rise to the alternately stacked plate-like structures. Figure 2 shows the microstructural variations of eutectics frozen at different rates. The left hand side of the figure represents the longitudinal and the right hand side the transverse section. It is clear from these figures that when the rate of solidification is decreased, the θ phase on the transverse section is grossly bent, appearing like a maze in the labyrinth, but that as the rate is increased, these bent plates tend to straighten out, resulting in the formation of a plate-like morphology. The measurements of the interlamellar spacings show that as the rate of solidification is increased, the lamellar spacings are decreased (figure 3). When lamellar spacings and the rate of solidification are plotted on a logarithmic scale, it was found that the exponent, n , in the form of $\lambda n V = \text{constant}$, was 2, in agreement with Jackson-Hunt's (1966) relationship, whose model has been developed on the basis of the volume diffusion of atoms in front of the solid-liquid interface, i.e. the relationship: $\lambda^2 V = 62.82$ was obtained for Al-Cu eutectics. Ample observations for $n = 2$ have been found in many other eutectic cases (Turnbull 1955) validating the so-called Jackson-Hunt model, $\lambda^2 v = \text{constant}$.

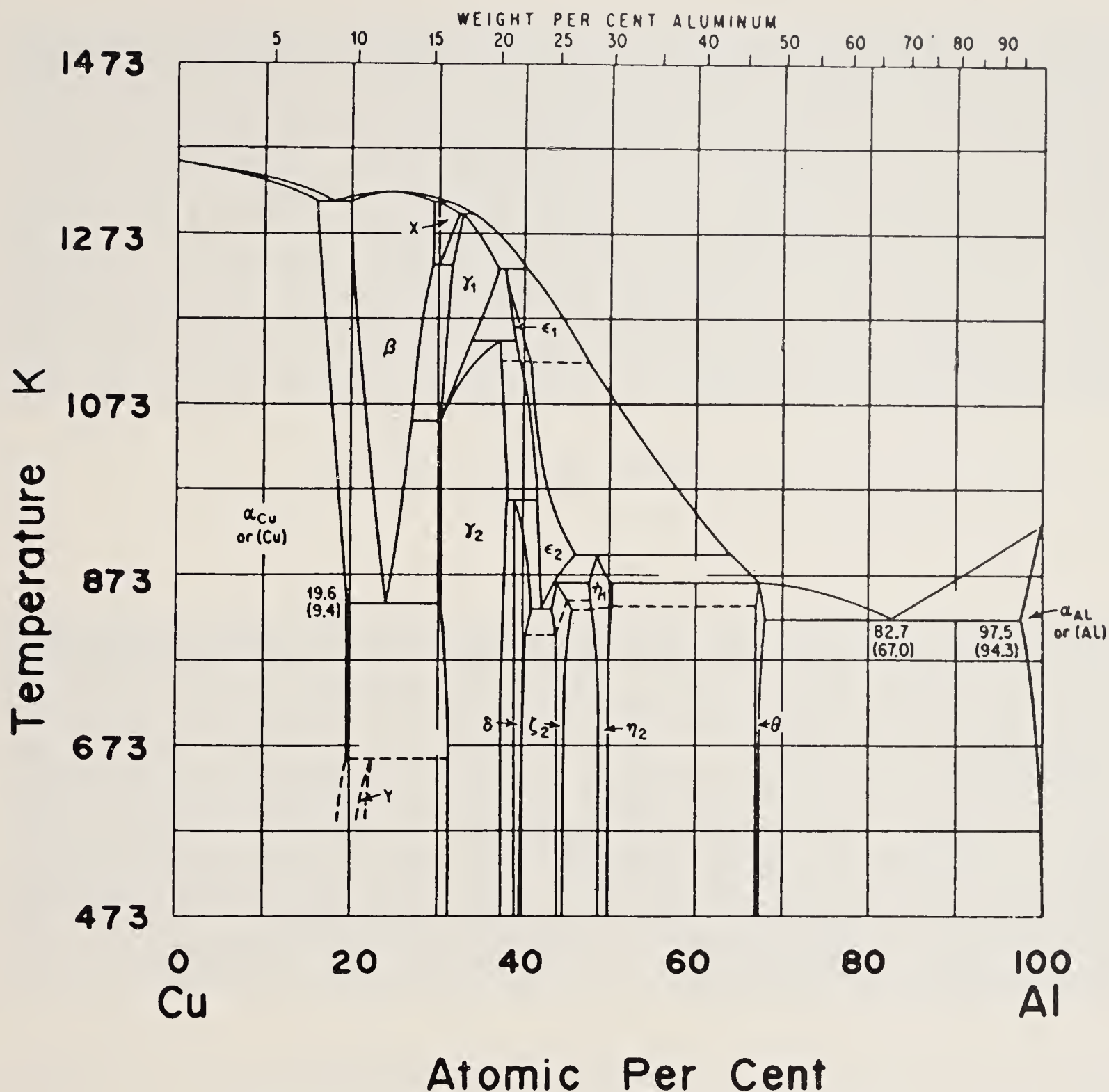
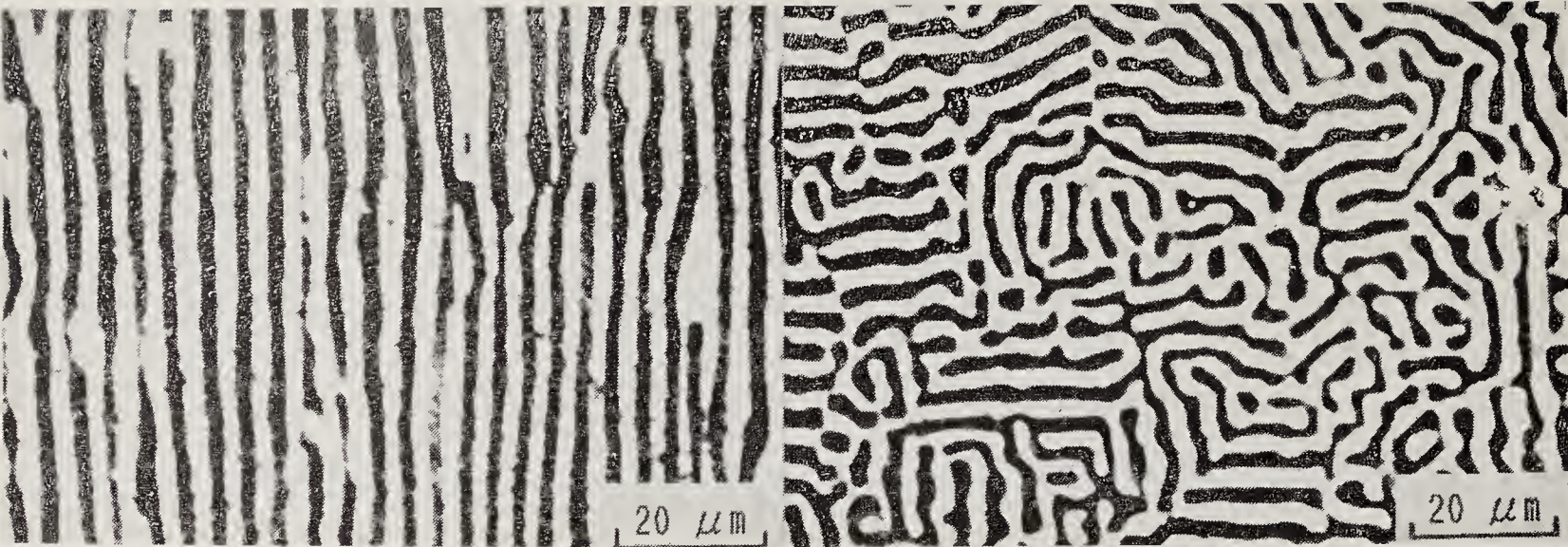


Figure 1. Equilibrium phase diagram for Al-Cu alloy system.

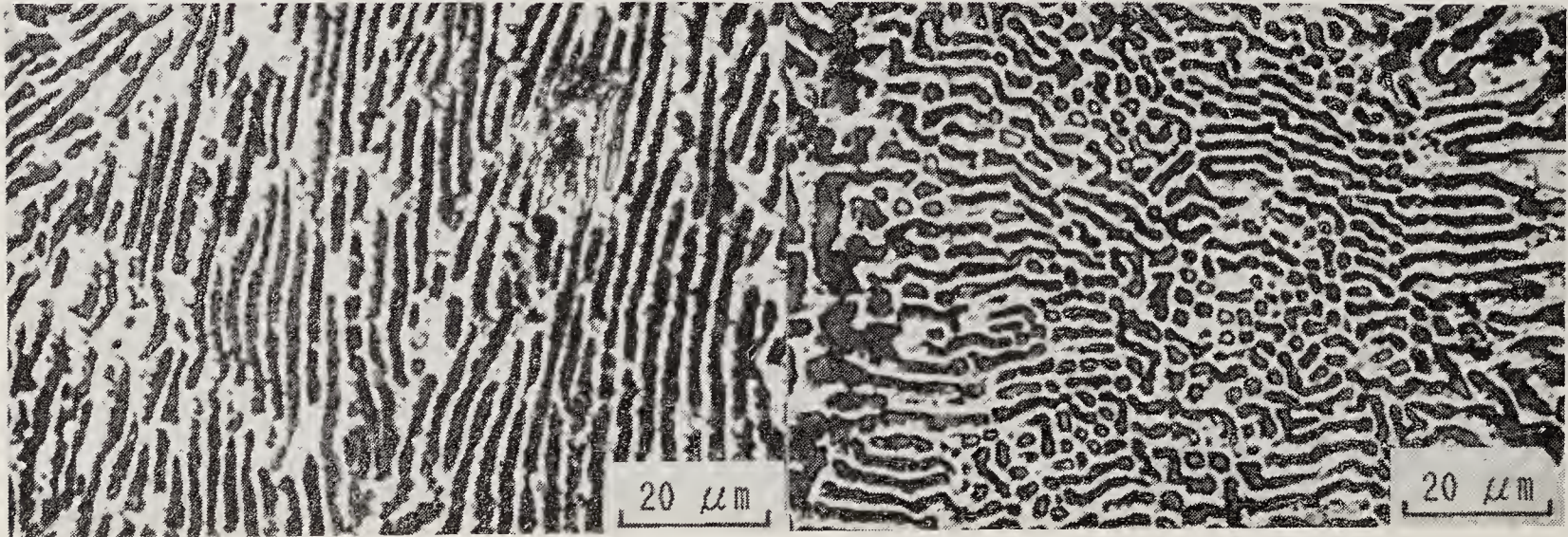
3.2 Cu-24.0 at.% Al eutectoidal alloys

The morphology of reaction products obtained through the eutectoidal transformation $\beta \rightarrow \alpha(\text{Cu}) + \gamma$ is similar to those found in the eutectic reaction. Figure 4 shows the plate-like morphology of Cu-Al eutectoidal products. It should be noted that as expected from the great difference in the diffusivities of atoms involved, the rate of solidification which is sufficient to produce a lamellar and well-ordered structure is much less in the eutectoidal reaction than in the eutectic reaction, although the temperature levels of these transformations are not very different. Thus, this difference is thought to result purely from the difference in the diffusivities of atoms in the liquid accumulated in front of the interface in the case of eutectics and in the solid accumulated in front of the interface in the case of eutectoids.

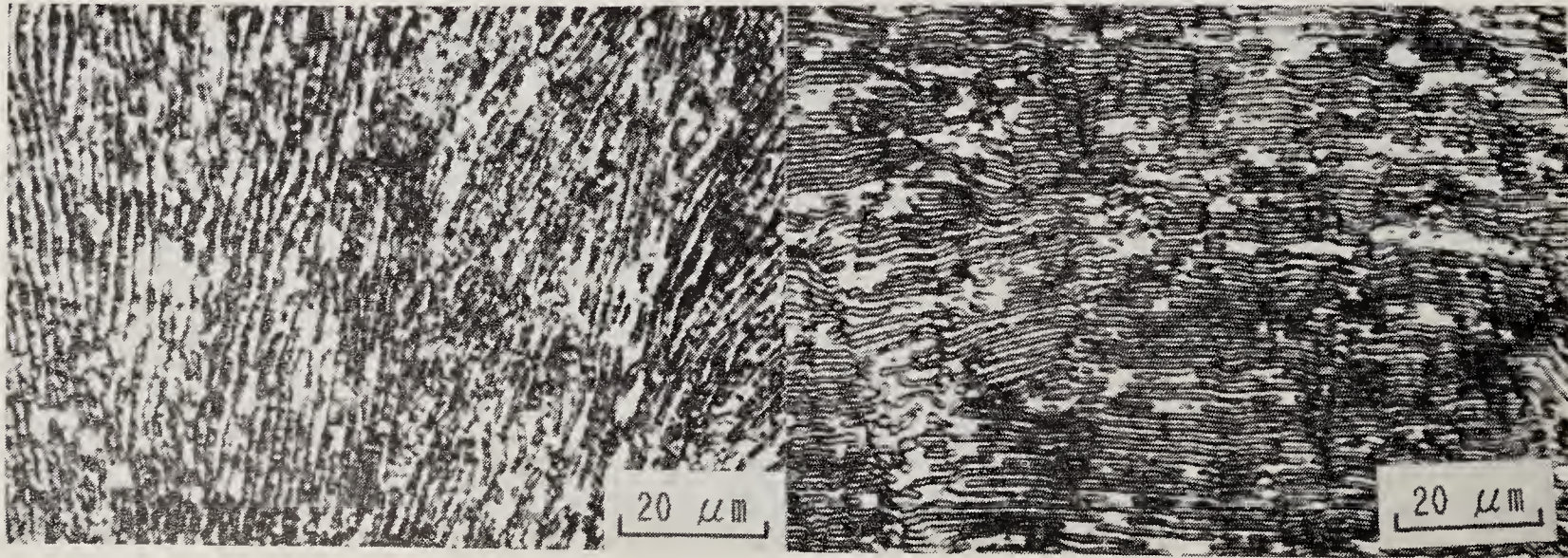
Interlamellar spacings as a function of the growth rates as shown in figure 5 indicate that the value of the exponent, n , in the relation $\lambda^n v = \text{const}$, is close to 4. This is in agreement with the model derived by Carpay *et al* (1972). They also found this in other



$$V = 2.8 \mu\text{m/s}$$



$$V = 9.3 \mu\text{m/s}$$



(a)

(b)

$$V = 33.3 \mu\text{m/s}$$

Figure 2. Eutectic structure of specimens cooled at different rates. (a) Longitudinal, and (b) transverse sections.

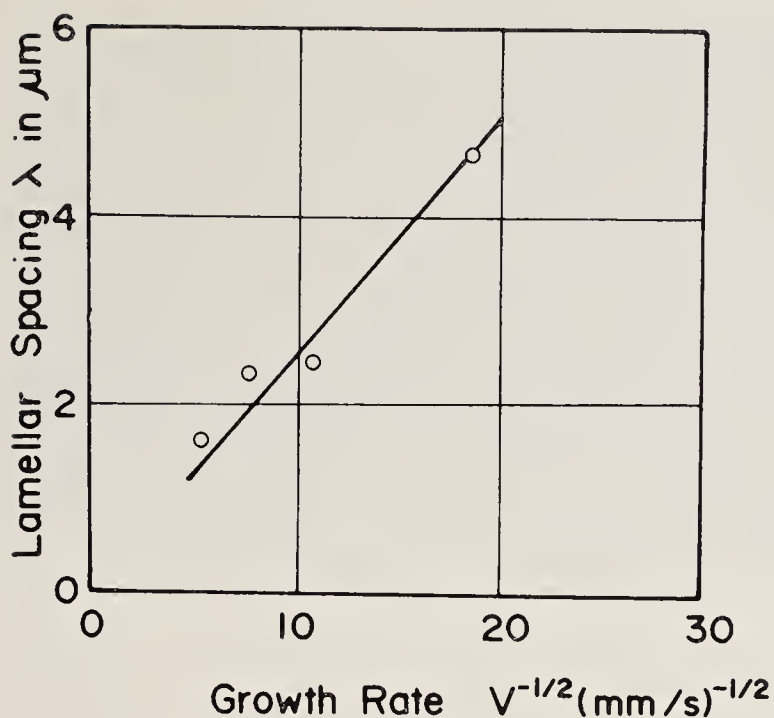


Figure 3. Relationship between lamellar spacings and growth rates for eutectic Al-Cu.

eutectoid systems such as Ni-In or Cu-In alloys. The following relationship was obtained in this investigation:

$$\lambda^4 v = 2.32 \times 10^{-2} (\mu\text{m}^5/\text{s}),$$

whose constant on the right hand side is about twice as great as the value obtained for the same alloy system by Carpay *et al* (1972).

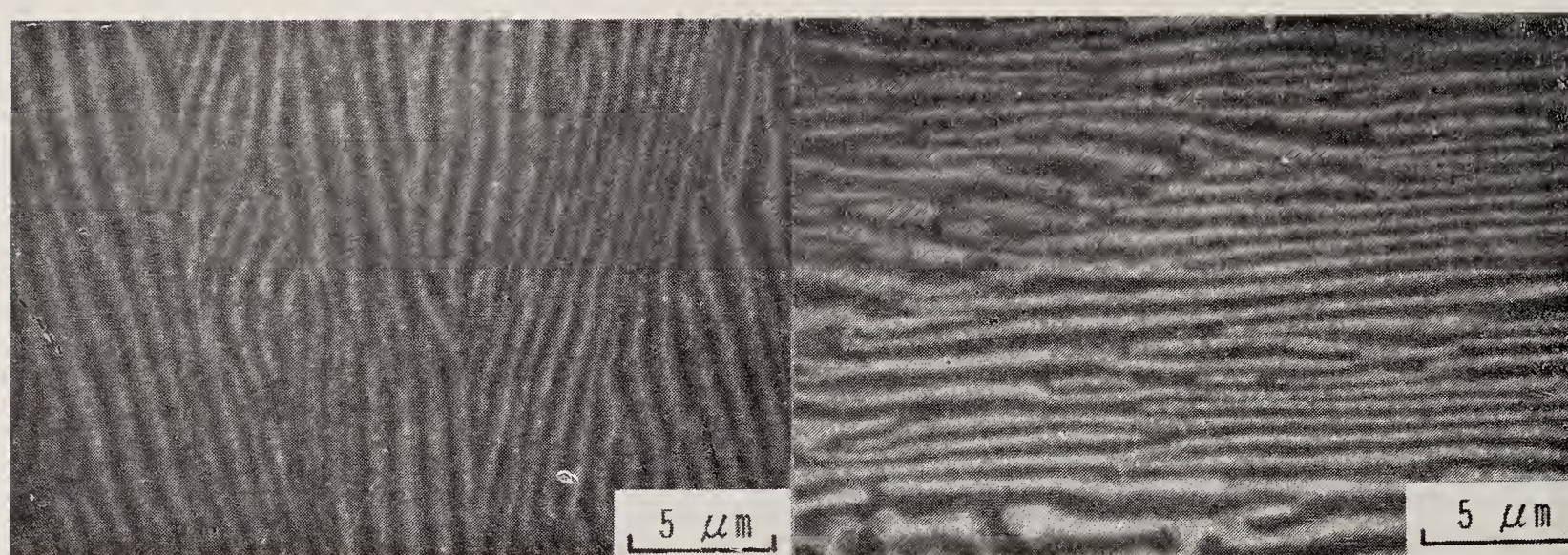
Transmission electron microscopy examinations have shown that the colonial structure, often seen in the eutectic specimen, was not observed in the eutectoidal specimen.

3.3 Discussions

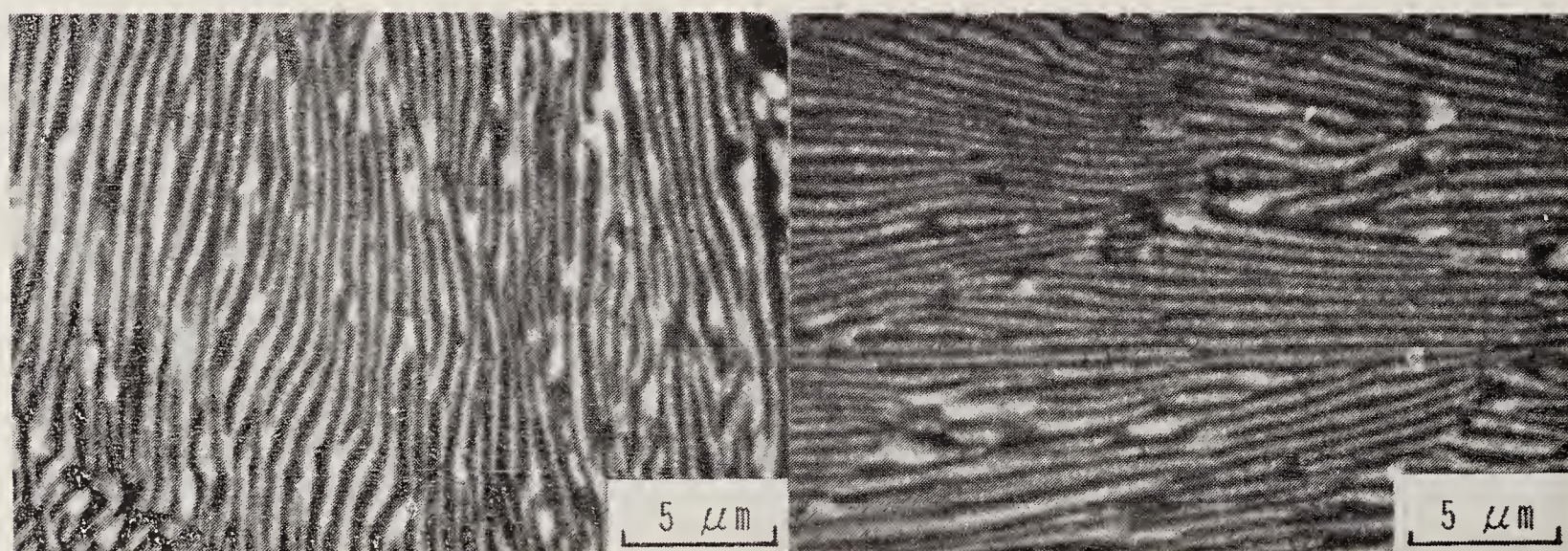
Several models on the phase separable transformations have been developed and many of them, under steady state conditions, suggested the form of $\lambda^n v = \text{constant}$ with different mechanisms ending up with different n -values. In table 1 are listed the n -values for various models previously proposed by other investigators.

The difference in n -values between eutectic and eutectoid reactions must now be taken into account. The basic difference involved in the production of similar morphology of the separated phases in the eutectic or the eutectoid Al-Cu system may be considered in the following steps:

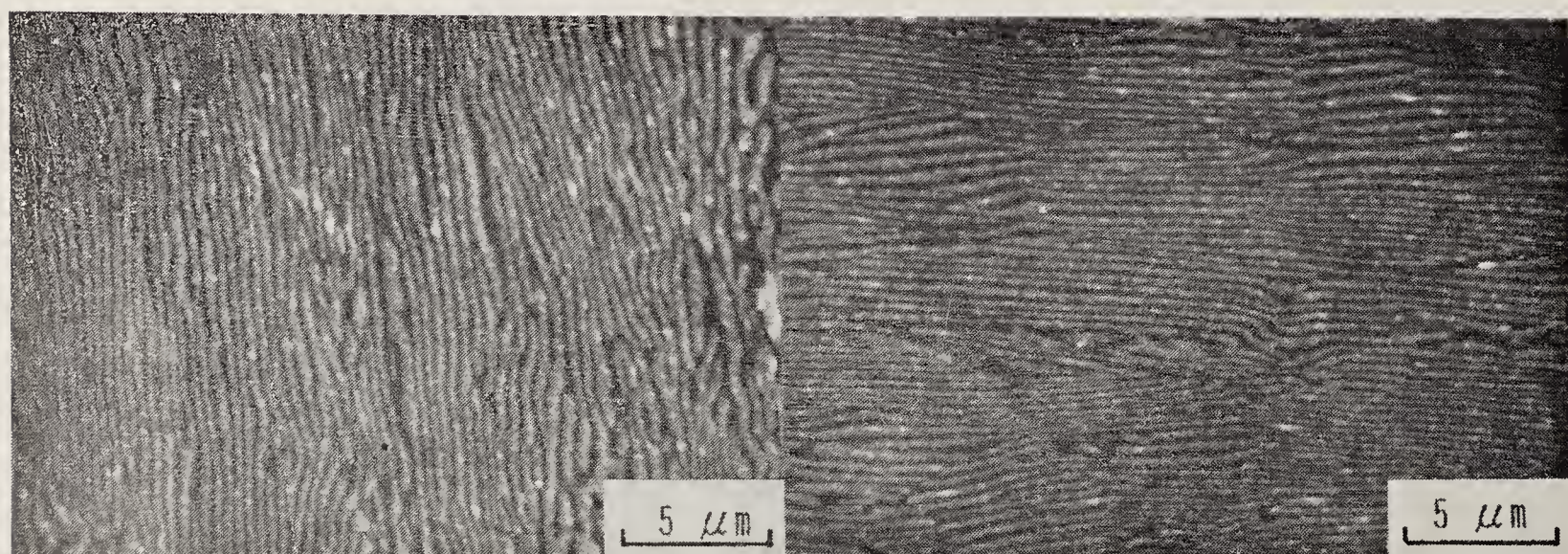
- (i) The critical difference is the state of the mother alloy, liquid in the case of the eutectic, and solid in the case of the eutectoid.
- (ii) Both cases involve the separation of two distinctly different phases and, therefore, the diffusion of Al or Cu atoms must occur at or near the boundaries.
- (iii) There is a compositional difference: the matrix phase of the eutectic is Cu atoms and that of the eutectoid is Al atoms, although the interdiffusion coefficient of Al atoms in Cu or Cu atoms in Al in the solid state will be of the same order of magnitude.
- (iv) The crystallographic orientation relationships in the product phases are different. This is simply due to the entirely different crystals in contact. The factors (i) and (ii) stated above seem to be critically a dominant factor resulting in the big difference in the rates of cooling suitable for obtaining well-aligned structures with parallel orientations. It is believed that the basic disparity arises due to whether the parent phase is liquid or



$$V = 0.4 \mu\text{m/s}$$



$$V = 1.5 \mu\text{m/s}$$



(a)

$$V = 2.9 \mu\text{m/s}$$

(b)

Figure 4. Eutectoid structure of specimens cooled at different rates. (a) Longitudinal and (b) Transverse sections.

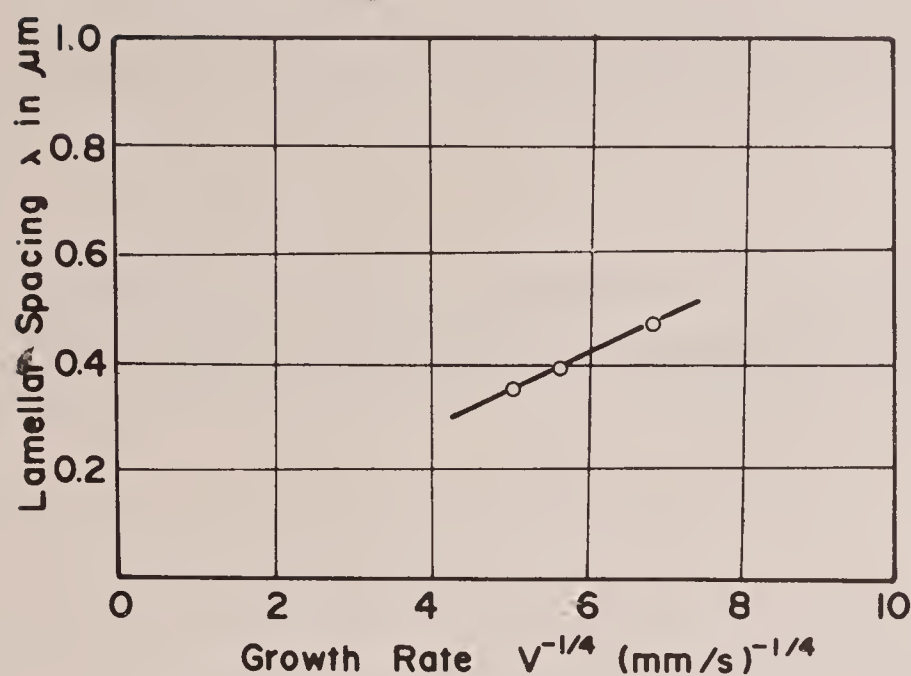


Figure 5. Relationship between lamellar spacings and growth rates for eutectoid Al-Cu.

solid, causing a big difference in the rate of atomic diffusion or in the drift due to fluid convection.

Taking the theoretical treatments available into account, the most suitable one for eutectoidal reaction is that of Carpay (1972) which is based on the boundary diffusion and which is highly possible in the solid state, and for the eutectic reaction is that of Jackson & Hunt (1966) which is based on volume diffusion which is quite plausible in the liquid state.

Carpay's (1972) treatment has turned out to be valid for several eutectoidal systems such as Co-Si, Ni-In and Cu-In alloy systems but did not work for Fe-Al and Al-Zn alloy systems.

4. Conclusions

The following conclusions are drawn from this investigation:

- (i) Purely from the values of the exponent, n , for $\lambda^n v = \text{constant}$, it was found that the eutectic reaction is controlled by volume diffusion of atoms accumulated in front of the solid-liquid interface.
- (ii) Similarly, the value of n for eutectoidal reaction $\lambda^n v = \text{constant}$ was $1/4$, which agrees with the model of Carpay (1972). It was believed that the controlling step of the eutectoid reaction was the diffusion of atoms along the solid-liquid boundaries.
- (iii) Combination of these results with those of previous investigations demonstrates

Table 1. Theoretically predicted exponent, n , for various growth models

Model	n -Values	Reference
Volume diffusion	1	Zener (1946)
	2	Jackson & Hunt (1966)
Boundary diffusion	2	Turnbull (1955)
	3	Shapiro & Kirkaldy (1968)
	4	Carpay (1972)

that the volume diffusion in the liquid is the governing step for eutectics, and that the boundary diffusion in the solid-liquid interface is the governing step for eutectoids in many other alloy systems.

The authors are grateful to Light Metals Foundation for financial support and Mita Industrial Co., Ltd. for supporting this research.

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Ultrasonic nondestructive evaluation of fibre-reinforced composite materials – a review

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Abstract. This paper reviews various ultrasonic nondestructive evaluation techniques applicable to fibre-reinforced composites. The techniques are briefly described and key references are cited. Methods to evaluate the reduced stiffness of composites due to micro-damage are described. Results show that for composites through-the-thickness attenuation increases and stiffness does not change due to transverse cracks, but in-plane stiffness and attenuation changes are substantial and can be measured by the Lamb wave techniques.

Keywords. Ultrasonics; nondestructive evaluation; composites; thin laminates.

1. Introduction

The excellent strength-to-weight ratio and the flexibility in tailoring the strength and stiffness of fibre-reinforced materials have made composites an indispensable structural material. Mixing of brittle but high strength fibres (e.g. graphite fibre, $\sigma_u = 2.5 \text{ GN/m}^2$) with viscoelastic and low strength (e.g. epoxy $\sigma_u = 0.10 \text{ GN/m}^2$) matrices, however, has created some very complex damage mechanism problems. Damage initiation and propagation are very different in composites from those in metals. The effect of mechanical, thermal or humidity loading is very complex in composites. Nondestructive evaluation (NDE) of in-service components is important because the damage initiation and growth mechanisms are not fully understood.

Various NDE techniques such as X-ray radiography, dye-penetrant tests, ultrasonics, thermography, acoustic emission, holography etc. have been successfully used to characterize damage in composites. This review is restricted to ultrasonic NDE of composites. It will also cover the technique of acoustic emission as well as the more recently developed method of acousto-ultrasonics.

In metals when damage is initiated, it becomes the nucleating site for further damage growth. On the other hand, Reifsnider *et al* (1983, pp. 399–420) have shown that in fibre-reinforced composites, a very different phenomenon takes place. Invariably, the first mode of damage in composites is matrix cracking. The fibres, being much stronger than the matrix, are able to carry the extra load due to the redistribution of stresses in

the vicinity of the matrix cracks. Fibres also act as matrix crack arresters. Stresses in the vicinity of the first crack are relieved and the next crack forms where the stresses again develop to a critical value. As a result the entire structure develops microcracks without endangering the integrity of the structure. Up to this stage of damage, identification of individual cracks does not serve any useful purpose. But due to these cracks the stiffness of the structure is reduced and hence the interest lies in the determination of the residual stiffness and the attenuation of the material. The transverse cracks in the 90° -plies are arrested by the 0° -plies. The point of crack arrest also becomes a point of stress concentration and, therefore, a nucleating site for the delaminations. At this stage of damage development microcracks have lost their significance and the attention is turned to the detection of macro-damage. The damage size and location are also of interest during manufacturing since any foreign material, such as dust, grease or oil left on the surface of the lamina during fabrication can result in debonding. Thus all NDE techniques can broadly be categorized as (1) techniques for detection, sizing and location of damage, and (2) evaluation of mechanical properties such as stiffness and ultrasonic attenuation.

2. Detection of damage, size and location

According to Krautkramer & Krautkramer (1983), Sokolov (1929) used a through-the-thickness transmission technique for flaw detection in metals. In regions where cracks exist, the sound intensity is small compared to the undamaged regions. Firestone (1945) utilized the pulse-echo method for the detection of flaws in materials. The ultrasonic pulses reflected from the flaws are detected to map out the flaws. The time taken for the waves to travel from the transmitting transducer to the receiving transducer gives the depth of the flaw if the wavespeed in the material is accurately known. Today, using

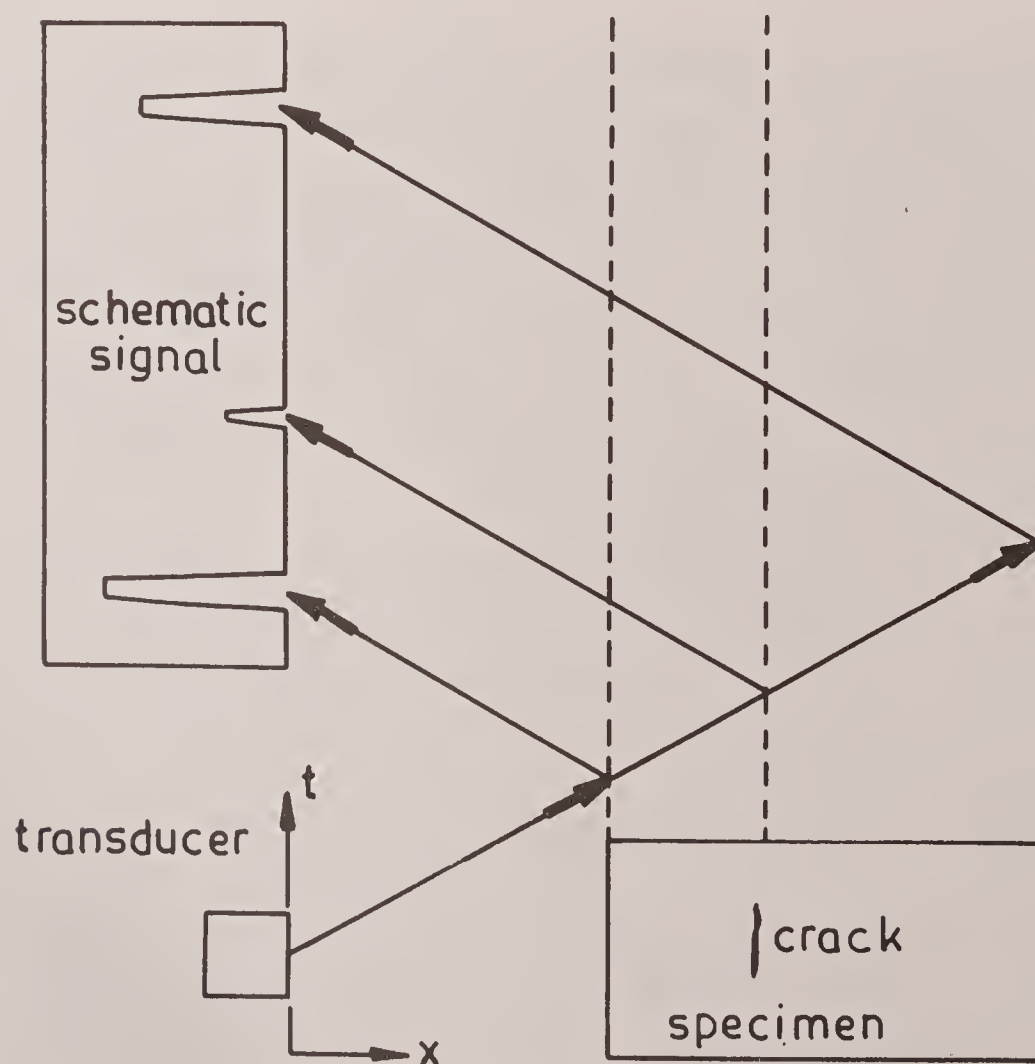


Figure 1. Schematic of the C-scan principle. The signal reflected from the crack is seen as a smaller signal between the reflections from the front and back surfaces.

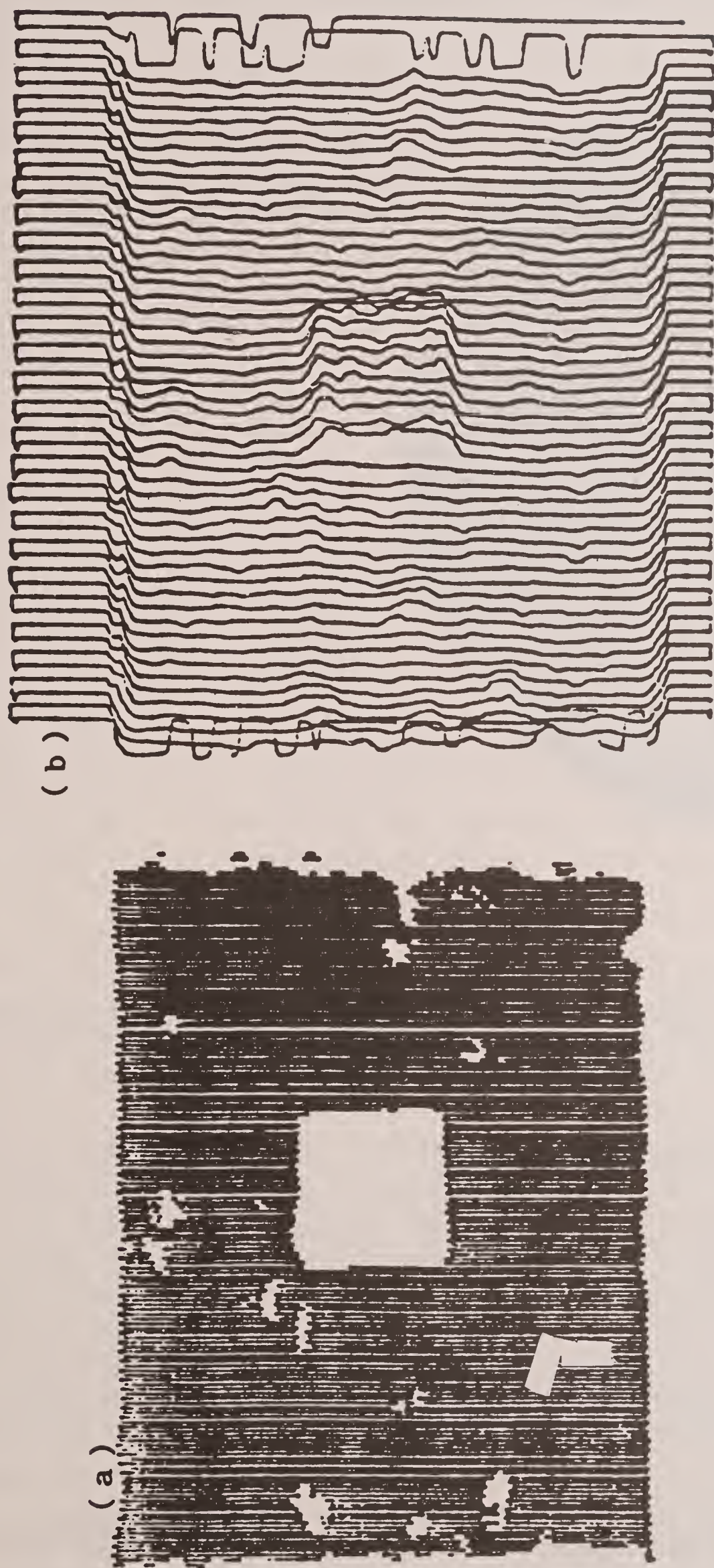


Figure 2. Representative C-scan of the graphite/epoxy laminate with an initial flaw in the form of an embedded inter-ply square film patch. (a) Pen-lift scan, (b) analog scan (Daniel *et al* 1981).

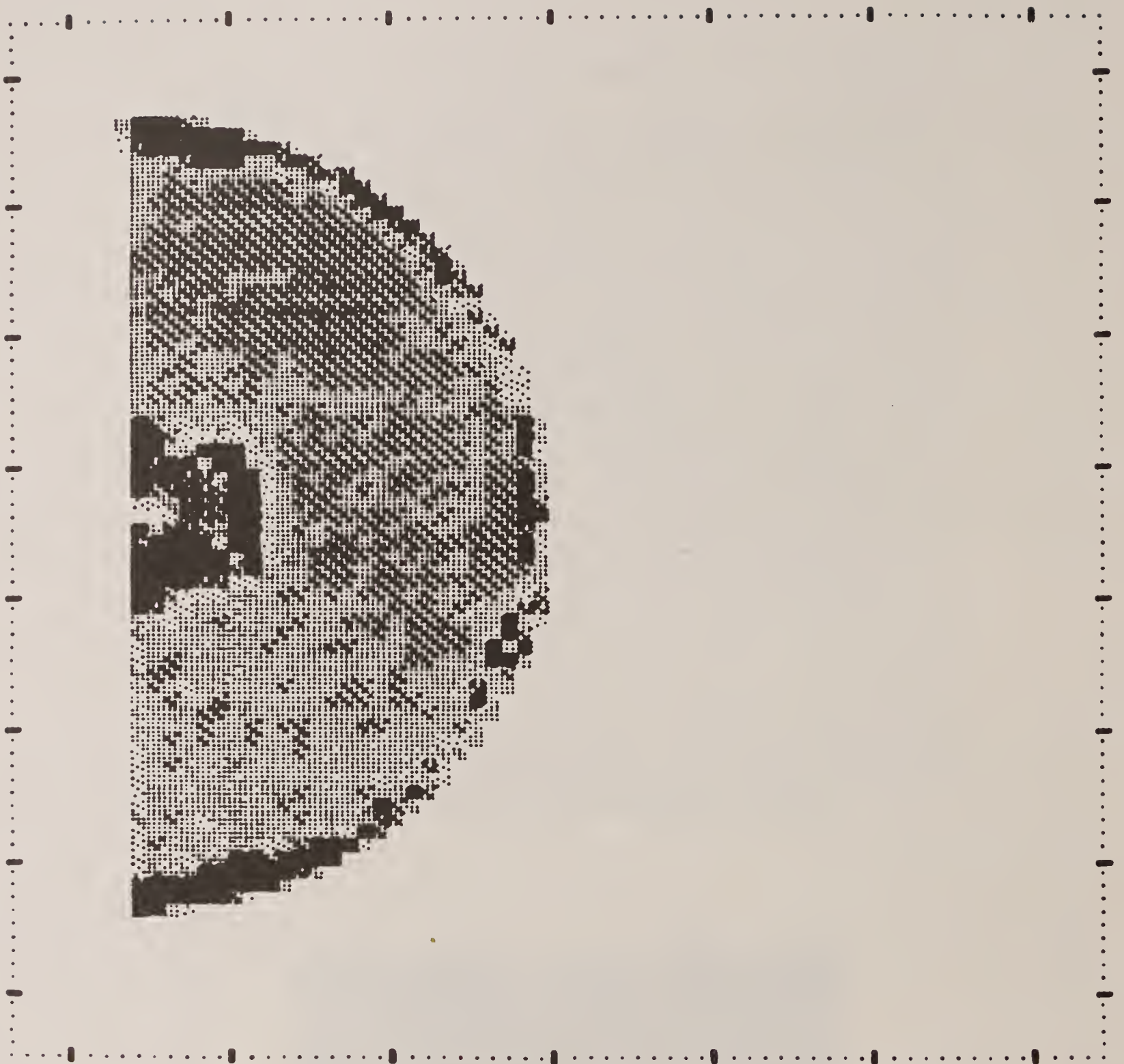


Figure 3. Digital C-scan of a 32-ply graphite/epoxy laminate with central and circumferential damage as well as thickness variation across the specimen (R A Blake, private commun.) (magnification = $0.8\times$; scan line spacing = 0.082 in.; scan time = 61 s).

computers with multicolored plotting facilities, a very detailed picture of the damage can be obtained. This pulse-echo technique is now better known as the “C-scan” technique. The basic concept behind this technique is shown in figure 1. The same transducer is used for production and reception of the waves. A typical signal trace is also shown. The first pulse is the front surface reflection, the second is reflection from the crack, and the third pulse is from the back surface. The presence of central pulse indicates a flaw in the material. The depth h of the crack can be obtained by an accurate measurement of time t and prior knowledge of the wavespeed c in the material: $h = ct/2$. Bar-Cohen *et al* (1979) have used this technique to detect 1 mm diameter delaminations in graphite/epoxy laminates with an accuracy of ± 0.2 mm in depth measurements. Daniel *et al* (1981) have used the C-scan for monitoring fatigue damage in composites; a typical C-scan picture from his work is reproduced in figure 2. Blake (1982, 1983) and Blake & Hartman (1984) have used computers for digital analysis and presentation of the damage pictures; see figure 3 for a digital C-scan picture.

Acoustic emission (AE) is another technique which is used for the detection of the onset of damage, its size and location. The deformation of a structure takes place under loading. This deformation eventually results in the generation of cracks or discontinuities in the structure. During the initiation and development of these cracks energy is released as sound waves. Thus the instant of initiation of damage can be recorded by detecting the AE waves. By a simple triangulation technique the location of damage can also be estimated fairly accurately. This is an excellent on-board technique, and although not a nondestructive technique, it is able to pinpoint the onset of failure.

Liptai (1972) found that in composites the acoustic emission takes place in two categories, the low level emission generated by plastic deformation of matrix and fibres, and the higher level emission generated by fibre failure, matrix cracking, interface failure and fibre pullout. Acoustic emission techniques have been used to study the real-time behavior of composite materials during fatigue loading by William & Reifsnider (1977). They have shown that AE data can be combined with video-thermography and dynamic recording of compliance to get meaningful results for the real-time NDE. Becht *et al* (1976) and Baily *et al* (1980) have used AE to investigate flaw formation and propagation in graphite reinforced plastic (GRP) pressure tubes, and to evaluate the fracture behavior of notched-bend specimens in flexural and fatigue experiments. They have reported some success in identifying fibre failure from matrix failure by amplitude distribution analysis. Arora & Tangri (1981) have used AE count rate (number of times the amplitude crosses the threshold) to estimate the growth rate of cracks in Zr-2.5% Nb. William & Egan (1979) and Belchamber *et al* (1983) have used spectral analysis of the AE signal to analyse fibre composite failure mechanisms. Since various types of damage may occur at the same time, it is rather difficult to differentiate between them. In this work, groups of AE events are treated as random data and are statistically analysed to identify group characteristics of different failure modes.

Vary & Bowles (1977, pp. 242–258) and Vary (1982) have combined AE with ultrasonics and developed the acousto-ultrasonic technique for characterization of fibre reinforced composites. The acoustic emission is stimulated by passing ultrasonic waves in structures. These AE waves are then detected by the usual AE procedures. These complex waves have been quantified by the “stress wave factor” (SWF) technique which will be described next. Talreja *et al* (1984, pp. 1099–1106) have used the technique developed by Vary to assess stiffness degradation in graphite/epoxy laminates.

The stress wave factor technique was proposed by Vary & Bowles (1977, pp. 242–258). The SWF is defined as; $SWF = g * r * n$, where g is the time interval over which the signal is recorded, r is the repetition rate of simulated AE waves and n is the number of oscillations each AE burst exceeds a fixed threshold voltage. Vary & Lark (1979) have shown that SWF can be correlated with the variations of the tensile and shear strengths of composite materials. William & Lampert (1980) have used a modified SWF for the degradation studies of impact damage in graphite-fibre composites. SWF is considered to be a measure of goodness of the material; a high SWF indicates a less damaged specimen. Obviously, SWF is inversely proportional to the attenuation in the material.

3. Acoustic parameters (wavespeed and attenuation) for the NDE of composites

It is well-known that the wavespeed of sound in a material is related to its stiffness:

$c^2 = E/\rho$ where c is the speed of sound, E is an approximate stiffness and ρ is the density. The presence of defects (e.g. voids, cracks, particles etc.) changes the effective stiffness of the material. When a mechanical wave of wavelength large in comparison to the crack size is propagated through such a medium, the change in stiffness is manifested as a change in the sound velocity according to the above equation. Furthermore, the defects act as wave scatterers. As a result, the defect population also manifests itself in the attenuation of the wave passed through the material. Kinra *et al* (1980, 1982), Kinra & Anand (1982), Kinra & Ker (1982, 1983) and Kinra (1984, pp. 983–991) have measured c and α (attenuation coefficient) for a variety of particulate composites. As expected both c and α were found to depend strongly on the volume fraction of inclusions. An unexpected result was that near a critical frequency, c and α were also found to be highly sensitive functions of frequency. This was attributed to the excitation of the fundamental resonance of the particles or voids.

The anisotropy of composites can greatly complicate the interpretation of the received ultrasonic signals. Kriz & Ledbetter (1983) and Kinra & Eden (1984) have obtained the solution of the Christoffel's equations given by Musgrave (1970) to graphically depict stiffness, longitudinal wave velocity and shear velocity variations in graphite/epoxy laminates. Some results obtained by Kinra & Eden (1984) are shown in figure 4. Here, slowness is the inverse of velocity. Shown also on the plot are the group velocity vectors (energy propagation directions) which are always perpendicular to the slowness surface.

Various researchers have used ultrasonics for the NDE of composites by relating the damage to the acoustic parameters. Ultrasonic waves have been used to measure the stiffness parameters in composites by Tauchert & Guzelsu (1971, 1972) Kriz & Stinchcomb (1979), Mann *et al* (1980), William *et al* (1980a) and Ueda *et al* (1983). Reynold & Wilkinson (1979) have used the measurement of difference in wave velocities for the estimation of porosity in composite materials. Heyman and Cantrell (1979, pp. 45–56) used a phase-insensitive transducer to study the effect of material inhomogeneity on ultrasonic measurements. Ultrasonic attenuation has been used to characterize damage in fibre composites by Hayford & Henneke (1979, pp. 184–200), Lee & Williams (1980), William *et al* (1980b, 1982), William & Doll (1980), Ulman &

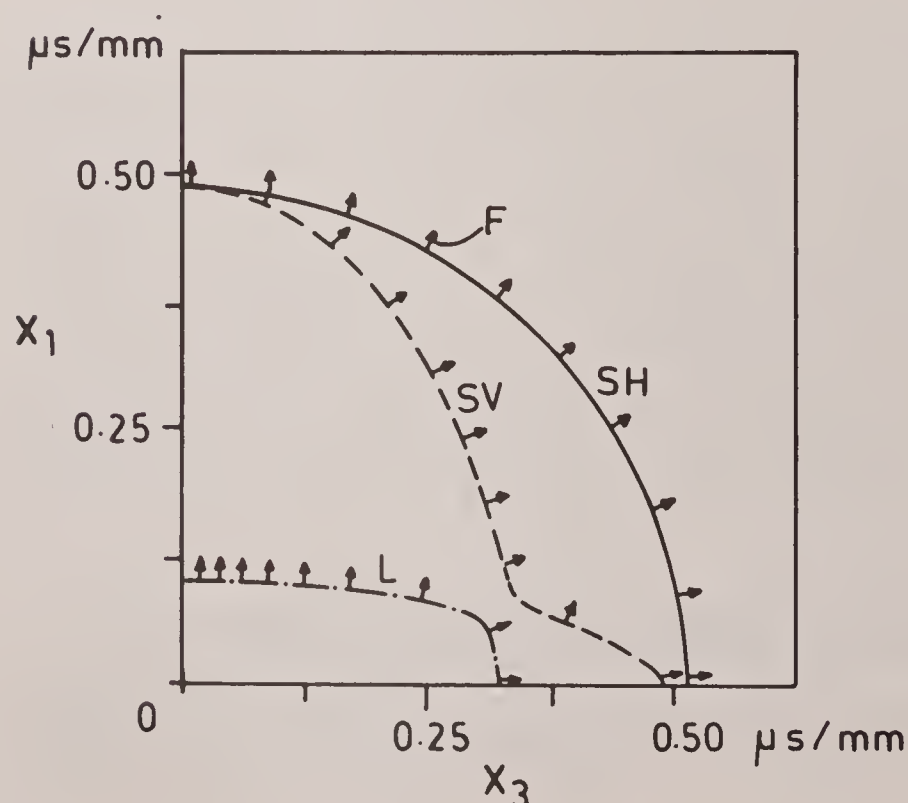


Figure 4. Slowness surfaces for a unidirectional graphite/epoxy composite. L: quasi-longitudinal, SV: quasi-shear (vertical), and SH: quasi-shear (horizontal) surfaces. F is the energy propagation direction.

Henneke (1982, pp. 323–342) and Hale & Ashton (1985). Ringermacher (1980, pp. 957–960) has utilized the changes in wavespeed to characterize damage in composites. He has shown that wavespeed is not a useful parameter to characterize damage in fibre composites. Cantrell *et al* (1980, pp. 1003–1005) have measured both attenuation and wavespeed and reached the same conclusion that while attenuation is a very sensitive measure of damage, wavespeed or stiffness is not. Eden (1985) has tested transverse cracks generated in $[0_6 90_4 0_2]_s$ graphite/epoxy specimens under static loading. He has used the pulse-echo spectroscopic technique to measure both wavespeed and attenuation. He has shown that as the damage increases, the pulses become broad (dispersive effect) and it becomes increasingly difficult to make good measurements of the acoustic parameters. He has also shown that when the waves propagate normal to the plane of the plate then the interaction between the cracks and waves is small. Hence wavespeed does not change but the increase in attenuation is appreciable.

4. Research of the present authors

We now summarize the principal research carried out by us. A detailed literature survey revealed that there was no method available to measure the acoustic parameters for thin specimens. We expect that aerospace structures will be thin and so Dayal *et al* (1986, pp. 899–904) have developed ultrasonic techniques for the measurement of acoustic parameters in thin specimens. When the specimen is thin the wave reflections from the two surfaces interfere and the classical time-of-flight method breaks down. In our technique the data is transformed from the time domain to the frequency domain by the use of fast fourier transforms (FFT). The techniques are fully computer-controlled and hence can be very easily adapted for automation and remote control. These techniques have been applied to the monitoring of damage in fibre-reinforced composites by Dayal & Kinra (1986). We found that attenuation is a very sensitive parameter of damage while wavespeed is rather insensitive. Results also show that the technique is very accurate and highly reproducible.

4.1 Through-the-thickness measurement of acoustic parameters

Let an infinite elastic plate be immersed in an elastic fluid (water). The time-versus-distance diagram of various reflected and transmitted pulses is shown in figure 5. If the plate is thick enough then 2 and 6 or 4 and 6 can be separated in the time domain. The available techniques (toneburst, pulse-superposition, pulse-echo etc.) are adequate to calculate the wavespeed and attenuation (though not very accurate for attenuation). If the plate is thin then 2, 6, 10 etc. or 4, 8, 12 etc. interfere and acoustic parameters cannot be calculated using any of the currently available techniques. The technique developed by us removes these restrictions and we have been successful in measuring wavespeed for aluminium plates down to 0.258 mm (10 mil) thickness. The equations used to reduce the data for these measurements are as follows. The first case we consider is when the plate thickness is such that a single pulse of signal is used and the various reflected or transmitted pulses can be separated in the time domain. Then,

$$(G^*/F^* - 1) = -T_{12} T_{21} \exp(-i2kh), \quad (1)$$

where G^* is the FFT of pulses 2 + 6 and F^* is the FFT of pulse 2; T_{12} is the transmission

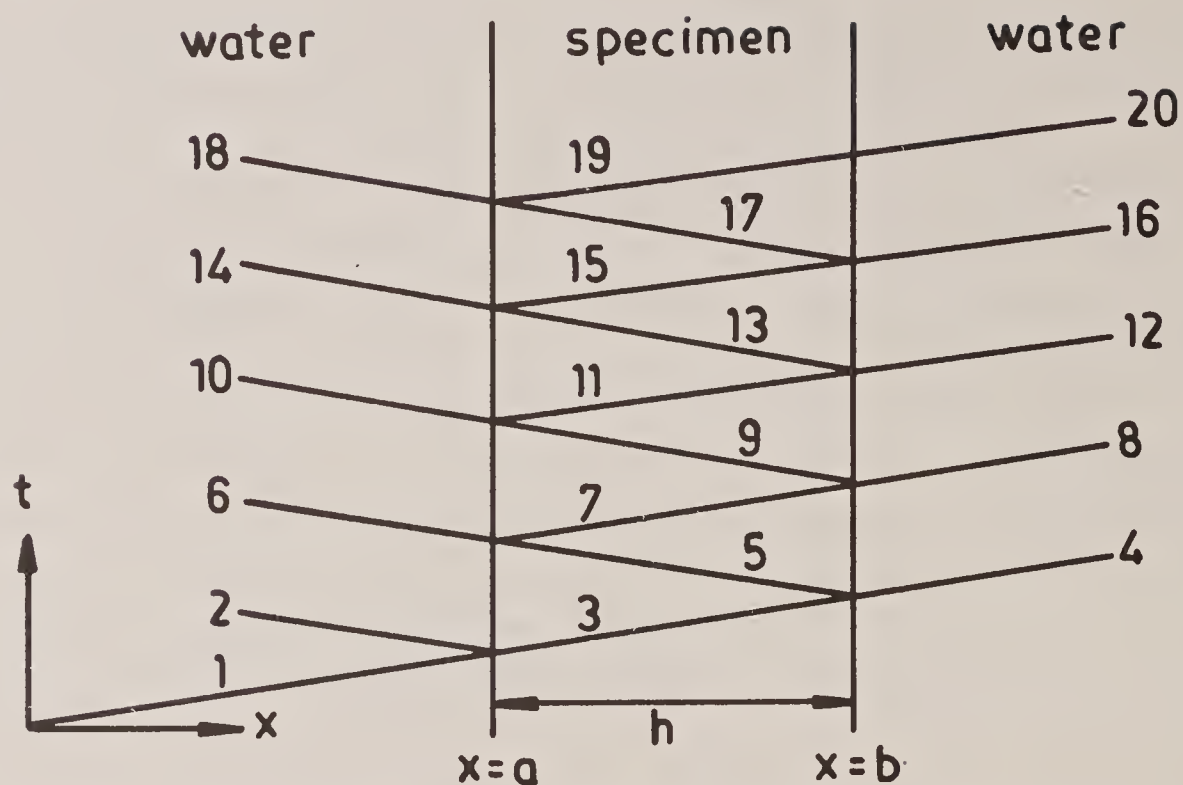


Figure 5. Time-versus-distance diagram for a plate immersed in a fluid.

coefficient for wave from water to plate and T_{21} is the transmission coefficient from plate to water; $k = k_1 + ik_2$ is the complex wavenumber, $k_1 = \omega/c$, ω is the circular frequency and c is the wavespeed, k_2 is the attenuation coefficient; and h is the plate thickness. Then,

$$c = 4\pi h / (-\phi/f) \quad \text{and} \quad k_2 = \ln M / 2h, \quad (2)$$

where ϕ is the phase of $(G^*/F^* - 1)$ and $M = |(G^*/F^*)/T_{12}T_{21}|$.

The wavespeed is calculated by plotting the phase-versus-frequency plot and the slope is obtained by fitting a least squares straight line through the points. Attenuation is obtained from the ratio of the magnitudes.

If the transmitted signal is used then the equations are,

$$G^*/F^* = R_{21}^2 \exp(-i2kh) \quad (3)$$

where G^* is the FFT of pulse 8 and F^* is the FFT of pulse 4.

The wavespeed and attenuation are calculated from (2) with $M = |(G^*/F^*)/R_{12}^2|$.

Now the case where the pulses cannot be separated is considered. If the reflected field is recorded by the transducer then the governing equation is

$$\beta/(1 + \beta) = R_{21}^2 \exp(-i2kh) \quad (4)$$

where $\beta = R_{12}R_{21}(G^*/F^* - 1)/T_{12}T_{21}$ and $R_{12}(R_{21})$ is the reflection coefficient of a wave in water (plate) from plate (water). G^* is the FFT of pulses $2 + 6 + 10 + \dots \infty$ and F^* is the FFT of a reference signal obtained by replacing the specimen by a thick plate of the same material and surface conditions.

If the transmitted signal is recorded by the transducer,

$$G^*/F^* = T_{12}T_{21} \exp\{-ih(k - k_0)\} / \{1 - R_{21} \exp(-i2kh)\} \quad (5)$$

where k_0 is the wavenumber of the wave in water, G^* is the FFT of pulses $4 + 8 + 12 + \dots \infty$ and F^* is the FFT of signal when there is no specimen between the transducers. Note that this is a quadratic equation in $\exp(-ikh)$.

These equations are equally valid for longitudinal and shear waves and have been used to measure both types of waves. They have been applied to a variety of specimens

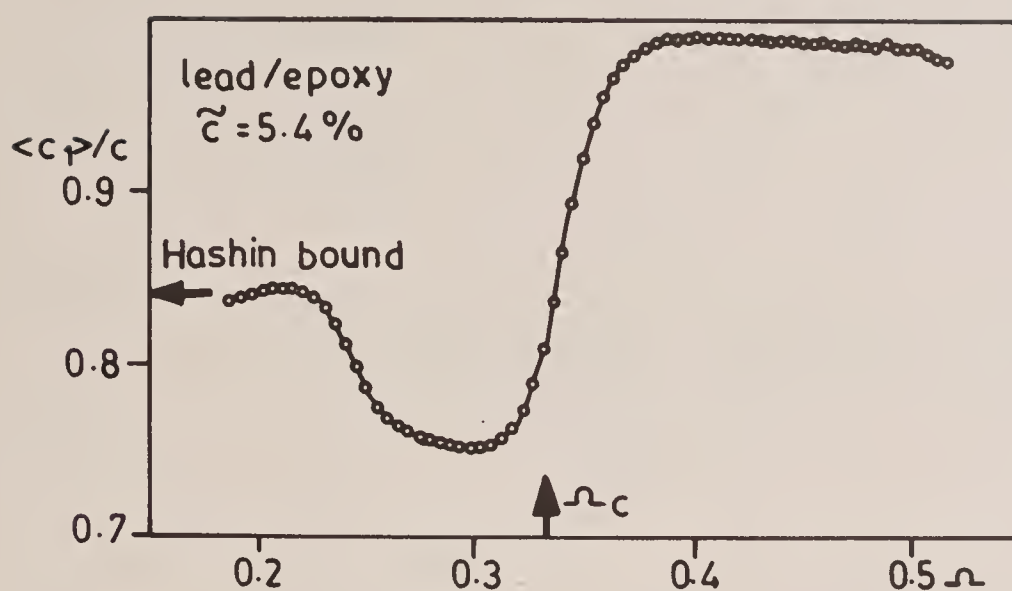


Figure 6. Normalized phase velocity as a function of frequency in a lead/epoxy particulate composite. Note large variations in velocity around the cut-off frequency Ω_c . Circles are digitized data points.

and the results are shown in table 1. The thickness of a plate of aluminium was reduced by careful machining and the wavespeed calculated at various thickness. The results show excellent repeatability and accuracy. It may be mentioned here that although the derivation assumed that the plate is elastic it is not difficult to show that these equations are rigorously valid for linear viscoelastic and dispersive materials provided attenuation is small, i.e., $k_2 \ll k_1$, which is generally the case. As an illustration of the efficacy of our technique in measuring k_1 and k_2 of highly dispersive and attenuative media, we tested random particulate composites consisting of lead spheres in an epoxy matrix. Figure 6 shows the phase velocity as a function of frequency in this specimen. Below the cut-off frequency, where the wavelength is large in comparison to the ball radius, the wavespeed is weakly dependent on frequency. Again at high frequencies, where the wavelength is small in comparison to the ball radius, the wavespeed is sensibly independent of the frequency. Around the cut-off frequency the wavespeed fluctuates very rapidly with increase in frequency. Here, cut-off frequency is the frequency that corresponds to the excitation of the rigid-body-translation resonance of the spheres. *It is emphasized that the entire dispersion curve was obtained in a single experiment.*

Next the results are presented for the NDE of fibre reinforced, graphite/epoxy, AS4/3502 composites. A $[0_6 90_4 0_2]_s$ laminate was tested. A static load was applied to the specimen and transverse cracks were produced in the 90° -plies. The test was interrupted at regular intervals and the number of cracks was determined by the edge replication technique. The specimen was then subjected to ultrasonic investigation. Figure 7 shows the results for tests at three different frequencies for attenuation as a function of applied loads. Shown also are the line sketches of the edge replications at different damage stages. A dramatic increase in attenuation is observed at all the frequencies. Figure 8 shows that for the same test there is practically no change in wavespeed. These results show that for the through-the-thickness measurements attenuation is a very good damage metric while wavespeed is not.

All the above measurements of wavespeed and attenuation have been made in the through-the-thickness direction where the crack-wave interaction is the weakest. This interaction is strongest when a wave travels perpendicular to the crack faces. This provided the motivation for examining the propagation of Lamb waves, which are described next.

4.2 Lamb wave technique for the NDE of composites

When Lamb waves are propagated in plates immersed in water, the displacement of the

Table 1. Longitudinal and shear wave speeds of different specimens.

Sample	Test frequency (MHz)	Thickness (mm)	c_1^a (mm μ s)	c_2^b (mm μ s)	$\sigma_c^c(\%)$	$\epsilon_c^d(\%)$	k_2	$\sigma_a^e(\%)$	Technique
Ni-based alloys	5	18.848	5.273	2.737	0.02	0.2	—	—	Sec/first
	5	6.365	5.877	3.163	0.02	0.2	—	—	Sec/first
	5	6.263	5.872	3.162	0.02	0.2	—	—	Sec/first
Epon	1	1.869	2.874	—	0.10	—	0.1340	1.4	All/first
	5	1.869	2.884	—	0.14	—	0.0940	1.5	All/first
	10	1.869	2.915	—	0.08	—	0.0975	1.0	All/first
	10	1.869	2.915	—	0.24	—	0.0979	2.2	Toneburst
Aluminium	10	2.807	6.357	—	—	0.2	—	—	Toneburst
	10	2.807	6.324	—	0.013	0.2	—	—	Sec/first
	10	2.807	6.327	—	0.010	0.2	—	—	All/first
	10	1.686	6.346	—	0.040	0.2	—	—	All/first
	10	1.001	6.354	—	0.030	0.2	—	—	All/first
	10	0.613	6.359	—	0.130	0.2	—	—	All/first
	10	0.256	6.323	—	0.140	0.2	—	—	All/first

^a c_1 longitudinal wavespeed; ^b σ_c standard deviation in c ; ^c ϵ_c error in c ; ^d c_2 shear wavespeed; ^e σ_a standard deviation in attenuation.

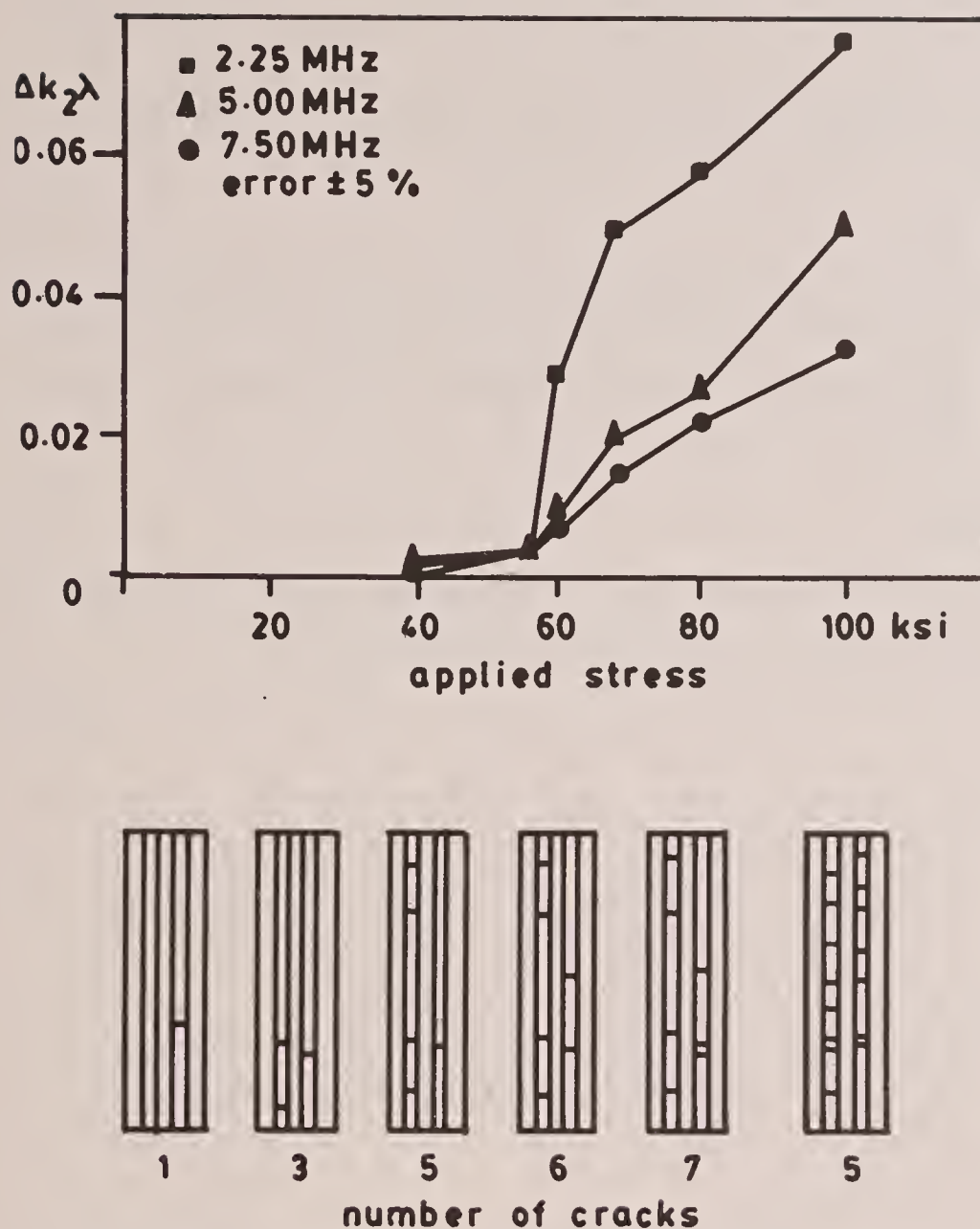


Figure 7. Attenuation increases dramatically with transverse cracks in a $[0_6 90_4 0_2]_s$ laminate at all three frequencies tested. The extent of damage and number of cracks are shown in the edge replication sketches.

two surfaces of the plate generates waves in water, hence the terminology “leaky Lamb waves”. Since these waves travel in the plane of the plate, in-plane stiffness governs the wavespeed of Lamb waves. Leaky Lamb waves were used by Bar-Cohen & Chimenti (1985) for the NDE of damage in composites. They have shown that various forms of damage can be identified by a null-zone measurement method. When a wave is incident upon a plate, it results in the excitation of a Lamb wave in the plate as well as a specular reflection. Due to phase change in the leaky wave, the specular reflection and the leaky wave interfere and a well-defined null zone is observed. The movement of the null zone has been related to the defects in their work. In our work we have adopted a different approach. The transducers are placed such that specular reflection is avoided and only

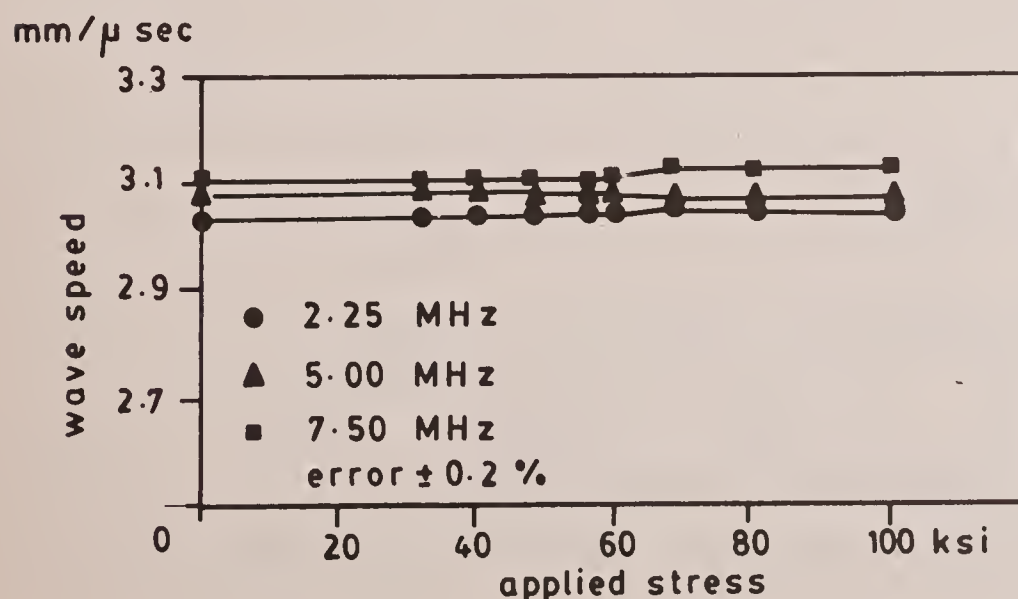


Figure 8. Longitudinal wavespeed remains unchanged with damage. Laminate is the same as in Figure 7.

the leaky Lamb waves are sensed. We measured the wavespeed and attenuation of the leaky Lamb waves.

The angle at which a Lamb wave is generated in the plate is governed by Snell's Law,

$$\sin(\theta_i)/\sin(\theta_r) = c_w/c_L, \quad (8)$$

where θ_i is the angle of incidence, $\theta_r = \pi/2$ is the angle of refraction, c_w is the wavespeed in water and c_L is the Lamb wavespeed. Habegar *et al* (1979) have shown by a rigorous analysis that at low frequencies (wavelength $\gg$ plate thickness) the Lamb wavespeed can be related to the in-plane stiffness (E_1) as

$$c_L^2 = E_1/\{\rho(1 - \nu_{12}\nu_{21})\} \quad (9)$$

where ν_{12} and ν_{21} are the two Poisson's ratios for the composite plate. Since for the cross-ply composites that we have tested, $\nu_{12}\nu_{21} \ll 1$, (8) reduces to

$$c_L^2 = E_1/\rho. \quad (10)$$

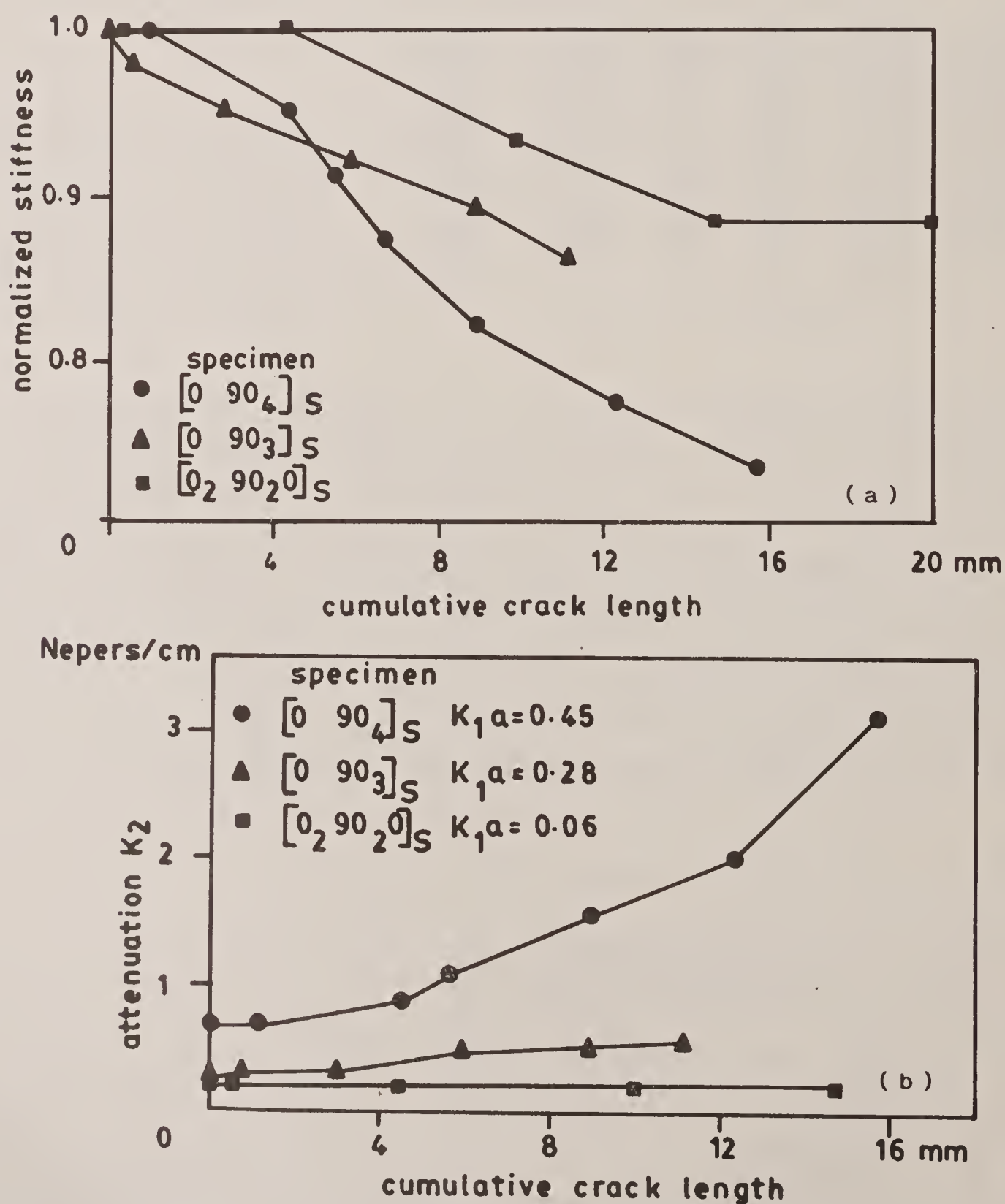


Figure 9. (a) Reduction of in-plane stiffness, and (b) increase in attenuation of leaky Lamb waves with damage, in three graphite/epoxy laminates.

Thus by the measurement of the Lamb wavespeed from (7) and substituting it in (9) the in-plane stiffness E_1 can be calculated. The results for a variety of graphite/epoxy laminates (Dayal & Kinra 1987) are reproduced here. Figure 9 shows the reduction in stiffness for three different laminate layups: $[0\ 90_4]_s$; $[0\ 90_3]_s$; and $[0_2\ 90_2\ 0]_s$. Let $2a$ be the length of the crack which is the same as the thickness of the contiguous 90° -plies. Let N be the linear density of the transverse cracks in the direction of wave propagation. In the absence of a more suitable measure of damage, we define cumulative crack length $L = 2aN$ and use it as a damage metric. Note that four 90° -plies contribute more to the overall stiffness of the laminate in comparison to three 90° -plies and hence their failure results in a larger stiffness reduction. The third laminate $[0_2\ 90_2\ 0]_s$ has four 90° -plies but they are divided in two and also the laminate has four 0° -plies. Thus the stiffness contribution of the 90° -plies is very low and hence it shows much lower stiffness reduction on damage. The increase in attenuation for the three laminates is shown in figure 9b. All tests were conducted at the same frequency of 0.5 MHz. The scattering cross-section of $[0\ 90_4]_s$ laminate is the largest ($k_1 a = 0.45$) and hence the attenuation increase is maximum. When $k_1 a \ll 1$, as for the $[0_2\ 90_2\ 0]_s$ laminate ($k_1 a = 0.06$), the wavelength is very large as compared to the crack length and the wave does not "see" the crack and the increase in attenuation is very low.

The results obtained by the authors thus show that for the through-the-thickness measurements the change in attenuation is a good measure of matrix cracking while wavespeed is not. But in the in-plane measurements both wavespeed and attenuation are sensitive measures of matrix cracks.

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